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L18: Entry 40 of 54 File: USPT Mar 2, 1993

DOCUMENT-IDENTIFIER: US 5191141 A
TITLE: Process for converting methanol to olefins using an improved metal aluminophosphate catalyst

Detailed Description Text (4):
The preparation of various ELAPOs are well known in the art and may be found in U.S. Pat. Nos.: 4,554,143 (FeAPO); 4,440,871 (SAPO); 4,853,197 (MAPO, MnAPO, ZnAPO, CoAPO); 4,793,984 (CAPO), 4,752,651 and 4,310,440, all of which are incorporated by reference. Generally, the ELAPO molecular sieves are synthesized by hydrothermal crystallization from a reaction mixture containing reactive sources of EL, aluminum, phosphorus and a templating agent. Reactive sources of EL are the metal salts such as the chloride and nitrate salts. When EL is silicon a preferred source is fumed, colloidal or precipated silica. Preferred reactive sources of aluminum and phosphorus are pseudo-boehmite alumina and phosphoric acid. Preferred templating agents are amines and quaternary ammonium compounds. An especially preferred templating agent is tetraethylammonium hydroxide (TEAOH).

Detailed Description Text (10):
The ELAPO molecular sieve of this invention may be used alone or they may be mixed with a binder and formed into shapes such as extrudates, pills, spheres, etc. Any inorganic oxide well known in the art may be used as a binder. Examples of the binders which can be used include alumina, silica, aluminum-phosphate, silica-alumina, etc. When a binder is used, the amount of ELAPO which is contained in the final product ranges from 10 to 90 weight percent and preferably from 30 to 70 weight percent.

Detailed Description Text (20):
16940 lbs. of water, 7700 lbs. of 75 wt % orthophosphoric acid, and 1200 lbs. of silica (HiSil.TM. 250) were added to tank A and mixed until homogeneous. 5400 lbs. of tetraethylammonium hydroxide (TEAOH, 40 wt % aqueous) were added to the mixture and blended in, followed by 5940 lbs. of di-n-propylamine (nPr.sub.2 NH). Finally, 4000 lbs. of alumina (Kaiser Versal.TM. 250) was added and the mixture was stirred until homogeneous. The resulting reaction mixture had an elemental composition in molar oxide ratios of:

Detailed Description Text (25):
99.8 grams of 85% orthophosphoric acid was combined with 95.5 grams of distilled water. 33.2 grams of a silica sol (Ludox.TM. LS, 30 wt % SiO.sub.2) was blended in, followed by 159.1 grams of a 40 wt % aqueous solution of tetraethylammonium hydroxide (TEAOH). Finally, 62.4 grams of a pseudo-boehmite form of precipitated alumina (Versal.TM. 250, 70.6 wt % Al.sub.2 O.sub.3, 29.4 wt % H.sub.2 O) was added and blended in. The resulting reaction mixture had an elemental composition in molar oxide ratios of:

Detailed Description Text (35):
101.1 grams of 85% orthophosphoric acid was combined with 110.4 grams of distilled water. 159.1 grams of a 40 wt % aqueous solution of tetraethylammonium hydroxide (TEAOH) was added to give a solution. With continuous stirring 8.4 grams of a silica sol (Ludox.TM. LS, 30 wt % SiO.sub.2) was blended in, followed by 68.7 grams of a pseudo-boehmite form of precipitated alumina (Versal.TM. 250: 70.6 wt %

Al.sub.2 O.sub.3, 29.4 wt % H.sub.2 O). The resulting reaction mixture had an elemental composition in molar oxide ratios of:

Detailed Description Text (40):
101.4 grams of 85% orthophosphoric acid was combined with 113.6 grams of distilled water. 161.8 grams of a 40 wt % aqueous solution of tetraethylammonium hydroxide (TEAOH) was added to give a solution. With continuous stirring 4.2 grams of a silica sol (Ludox.TM. LS, 30 wt % SiO.sub.2) was blended in, followed by 68.9 grams of a pseudo-boehmite form of precipitated alumina (Versal.TM. 250, 70.6 wt % Al.sub.2 O.sub.3, 29.4 wt % H.sub.2 O). The resulting reaction mixture had an elemental composition in molar oxide ratios of:

Detailed Description Text (45):
The catalysts prepared in Examples 1-5 were evaluated for the conversion of methanol to light olefins in a fixed bed pilot plant. A 10 gram sample in the form of 20-40 mesh agglomerates was used for the testing. Before testing, each sample was calcined in air in a muffle oven at 650.degree. C. for 2 hours and then pre-treated in situ by heating to 400.degree. C. for 1 hour under hydrogen. The pretreated sample was now contacted with a feed consisting of methanol, H.sub.2 O and H.sub.2 in a 1/4.6/5.3 molar ratio at 400.degree. C., 5 psig and 1 hr.sup.-1 MeOH WHSV. The composition of the effluent was measured by an on-line GC after 45 minutes on stream to determine initial conversion and selectivities. To determine catalyst life the runs were continued until methanol or dimethylether was observed in the reactor effluent. The results are tabulated in Table 1. This data shows that improved catalyst performance, in terms of reduced C.sub.3 by-product formation and/or increased catalyst life, is obtained by the use of a catalyst having an average particle size of less than 1.0 .mu.m and/or less than 0.05 moles fraction Si. Superior performance is obtained with catalysts that combine these two properties.

CLAIMS:

7. The process of claim 6 where the binder is selected from the group consisting of alumina, silica, aluminum phosphate, silica-alumina and mixtures thereof.

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L23: Entry 3 of 3 File: USPT Nov 16, 1982

DOCUMENT-IDENTIFIER: US 4359456 A
** See image for Certificate of Correction **
TITLE: Antiperspirant activity of basic aluminum compounds

Brief Summary Text (11):
In Izvestiya Akademii Nauk Latvinskoi SSR, Seriya Khimicheskaya, No. 4 (1973) 421-426, there is described an investigation into the effect of the time and the temperature of heating and the concentration of solutions of aluminium chlorhydrate Al.sub.2 (OH).sub.5 Cl on the preparation of boehmite. It is reported that boehmite is formed when aluminium chlorhydrate solutions with an aluminium ion concentration of 6.1 to 48.6 mg/ml are heated at 160.degree. C. for 5 hours under hydrothermal conditions. When the aluminium ion concentration is 64.8 mg/ml or higher it is reported that no boehmite is formed as indicated by X-ray crystallography or differential thermal analysis. Furthermore, results are given of a study of the effect of temperature on the heating of aluminium chlorhydrate solutions with an initial Al.sup.3+ concentration of 8.1 and 16.2 mg/ml in which study solutions were heated for 5 hours at 100.degree., 120.degree., 140.degree., 160.degree. and 180.degree. C. in sealed ampoules. At 100.degree. and 120.degree. C. the samples did not exhibit the characteristic reflections of boehmite in the X-ray diffraction diagrams.

Brief Summary Text (44):
The conditions of heat treatment described herein have been found to give rise to the improved basic aluminium compound in amorphous form, and in particular the formation of boehmite, as determined by X-ray diffraction, has not been observed. The production of a substantial amount of boehmite or other crystalline forms of alumina would be considered to be disadvantageous.

Brief Summary Text (67):
(B) a cosmetically acceptable adjunct selected from the group consisting of a perfume, thickener, alcohol and propellant,

Brief Summary Text (72):
In accordance with another aspect of the invention there is provided an antiperspirant composition comprising an aqueous solution of an antiperspirant active compound in combination with an adjunct which is a perfume, thickener, alcohol or propellant. The antiperspirant composition may be in the form of a lotion comprising an aqueous or aqueous alcoholic solution of the basic aluminium compound having an aluminium concentration of from 2.5 to 8.5% by weight and 0.1 to 5% by weight of a thickening agent. Suitable thickening agents for antiperspirant lotions are well known to those skilled in the art, and include for example, magnesium aluminium silicates. Thickening may also be effected by emulsifying an oil or the like in the composition. Furthermore, the composition may comprise an aqueous or aqueous alcoholic solution of the basic aluminium compound having an aluminium concentration of from 2.5% to 8.5% by weight and from 0.1 to 1% by weight of perfume.

Brief Summary Text (73):

The composition may comprise an aqueous alcoholic solution of the basic aluminium compound containing from 1 to 60% by weight of an alcohol. These aqueous alcoholic compositions preferably contain ethanol or isopropanol as the alcohol which are preferably present in an amount of from about 1% to about 30% by weight of the composition. Antiperspirant compositions comprising an aqueous solution of the active compound may contain from about 1 to 80% by weight of a propellant.

Brief Summary Text (74):
The antiperspirant composition may also comprise in combination a powdered antiperspirant active compound and a powdered inert solid diluent or organic liquid carrier. The composition may be in the form of a powder aerosol composition comprising a suspension of the basic aluminium compound in particulate form in a liquid carrier, said composition also comprising a propellant. In particular the composition may be in the form of a powder aerosol comprising:

Brief Summary Text (78):
D. from about 70% to about 96% by weight of a propellant.

Brief Summary Text (82):
The propellant can be a liquefied hydrocarbon, halogenated hydrocarbon or a mixture thereof. Examples of materials that are suitable for use as propellants are given in the above-mentioned patents and include trichlorofluoromethane, dichlorodifluoromethane, dichlorotetrafluoroethane, monochlorodifluoromethane, trichlorotrifluoroethane, propane, butane, 1,1-difluoroethane, 1,1-difluoro-1-chloroethane, dichloromonofluoromethane, methylene chloride, isopentane and isobutane, used singly or admixed. Trichlorofluoromethane, dichlorodifluoromethane, dichlorotetrafluoroethane, and isobutane, used singly or admixed, are preferred.

Brief Summary Text (83):
Examples of materials that are suitable for use as permanent gas propellants are nitrogen, carbon dioxide and nitrous oxide.

Detailed Description Paragraph Table (10):
_____ % _____
Treated powder 4.50 Isopropyl myristate 6.00 Aerosil 200 (pyrogenic silica) 0.45 Perfume 0.44 Propellant.sup.1 q.s. 100.00
_____ .sup.1 Propellant CCl.sub.3 F:CCl.sub.2 F.sub.2 50:50 by weight

Detailed Description Paragraph Table (11):
_____ % _____
Treated powder 3.50 Isopropyl myristate 8.00 Bentone 38 (hydrophobic clay) 0.60 Ethyl alcohol (95%) 0.27 Perfume 0.40 Propellant.sup.1 q.s. 100.00
_____ .sup.1 Propellant CCl.sub.3 F:CCl.sub.2 F.sub.2 :CClF.sub. 2 --CClF.sub.2 :nbutane 20:10:50:20 by weight

Detailed Description Paragraph Table (12):
_____ % _____
Treated powder 3.50 Dibutyl phthalate 8.00 Stearoyl monoethanlamide 0.60 Perfume 0.40 Propellant.sup.1 q.s. 100.00 _____ .sup.1 Propellant CCl.sub.3 F:CCl.sub.2 F.sub.2 :butane 40:30:30 by weigh

Detailed Description Paragraph Table (14):
_____ % _____
Treated powder 4.00 Bentone 38 (hydrophobic clay) 0.40 Isopropyl myristate 6.00 Perfume 0.50 Propellant 142b 89.10 _____

L18: Entry 19 of 54 File: USPT May 15, 2001

DOCUMENT-IDENTIFIER: US 6231749 B1
TITLE: Production of high viscosity index lubricants

Brief Summary Text (8):
Processes of this type, employing a waxy feed which is subjected to hydrocracking over an amorphous bifunctional catalyst such as nickel-tungsten on alumina or silica-alumina are disclosed, for example, in British Patents Nos. 1,429,494, 1,429,291 and 1,493,620 and U.S. Pat. Nos. 3,830,273, 3,776,839, 3,794,580, and 3,682,813. In the process described in GB 1,429,494, a slack wax produced by the dewaxing of a waxy feed is subjected to hydrocracking over a bifunctional hydrocracking catalyst at hydrogen pressures of 2,000 psig or higher, followed by dewaxing of the hydrocracked product to obtain the desired pour point. Dewaxing is stated to be preferably carried out by the solvent process with recycle of the separated wax to the hydrocracking step.

Detailed Description Text (16):
The metal component of the catalyst is supported on a porous, amorphous metal oxide support and alumina is preferred for this purpose although silica-alumina may also be employed. Other metal oxide components may also be present in the support although their presence is less desirable. Consistent with the requirements of a lube hydrocracking catalyst, the support should have a pore size and distribution which is adequate to permit the relatively bulky components of the high boiling feeds to enter the interior pore structure of the catalyst where the desired hydrocracking reactions occur. To this extent, the catalyst will normally have a minimum pore size of about 50 .ANG. i.e with no less than about 5 percent of the pores having a pore size less than 50 .ANG. pore size, with the majority of the pores having a pore size in the range of 50-400 .ANG. (no more than 5 percent having a pore size above 400 .ANG.), preferably with no more than about 30 percent having pore sizes in the range of 200-400 .ANG.. Preferred catalysts for the first stage have at least 60 percent of the pores in the 50-200 .ANG. range. The pore size distribution and other properties of some typical lube hydrocracking catalysts suitable for use in the first stage are shown in Table 4 below:

Detailed Description Text (18):
The catalyst activity may also be maintained at the desired level by in situ fluoriding in which a fluorine compound is added to the stream which passes over the catalyst in this stage of the operation. The fluorine compound may be added continuously or intermittently to the feed or, alternatively, an initial activation step may be carried out in which the fluorine compound is passed over the catalyst in the absence of the feed e.g. in a stream of hydrogen in order to increase the fluorine content of the catalyst prior to initiation of the actual hydrocracking. In situ fluoriding of the catalyst in this way is preferably carried out to induce a fluorine content of about 1 to 10 percent fluorine prior to operation, after which the fluorine can be reduced to maintenance levels sufficient to maintain the desired activity. Suitable compounds for in situ fluoriding are orthofluorotoluene and difluoroethane.

Detailed Description Text (27):
The support material for the paraffin hydroisomerization catalyst is zeolite beta, a highly siliceous, zeolite in a form which has the required low level of acid

activity to minimize paraffin cracking and to maximize paraffin isomerization. Low acidity values in the zeolite may be obtained by use of a sufficiently high silica:alumina ratio in the zeolite, achievable either by direct synthesis of the zeolite with the appropriate composition or by steaming or dealuminization procedures such as acid extraction. Isomorphous substitution of metals other than aluminum may also be utilized to produce a zeolite with a low inherent acidity. Alternatively, the zeolite may be subjected to alkali metal cation exchange to the desired low acidity level, although this is less preferred than the use of a zeolite which contains framework elements other than aluminum.

Detailed Description Text (28):
Zeolite beta is the preferred support since this zeolite has been shown to possess outstanding activity for paraffin isomerization in the presence of aromatics, as disclosed in U.S. Pat. No. 4,419,220. The low acidity forms of zeolite beta may be obtained by synthesis of a highly siliceous form of the zeolite e.g with a silica-alumina ratio above about 50:1 or, more readily, by steaming zeolites of lower silica-alumina ratio to the requisite acidity level. Another method is by replacement of a portion of the framework aluminum of the zeolite with another trivalent element such as boron which results in a lower intrinsic level of acid activity in the zeolite. The preferred zeolites of this type are those which contain framework boron and normally, at least 0.1 weight percent, preferably at least 0.5 weight percent, of framework boron is preferred in the zeolite. In zeolites of this type, the framework consists principally of silicon tetrahedrally coordinated and interconnected with oxygen bridges. The minor amount of an element (alumina in the case of alumino-silicate zeolite beta) is also coordinated and forms part of the framework. The zeolite also contains material in the pores of the structure although these do not form part of the framework constituting the characteristic structure of the zeolite. The term "framework" boron is used here to distinguish between material in the framework of the zeolite which is evidenced by contributing ion exchange capacity to the zeolite, from material which is present in the pores and which has no effect on the total ion exchange capacity of the zeolite.

Detailed Description Text (29):
Methods for preparing high silica content zeolites containing framework boron are known and are described, for example, in U.S. Pat. Nos. 4,269,813; a method for preparing zeolite beta containing framework boron is disclosed in U.S. Pat. No. 4,672,049. As noted there, the amount of boron contained in the zeolite may be varied by incorporating different amounts of borate ion in the zeolite forming solution e.g. by the use of varying amounts of boric acid relative to the forces of silica and alumina. Reference is made to these disclosures for a description of the methods by which these zeolites may be made.

Detailed Description Text (30):
In the present low acidity zeolite beta catalyst, the zeolite should contain at least 0.1 weight percent framework boron, preferably at least 0.5 weight percent boron. Normally, the maximum amount of boron will be about 5 weight percent of the zeolite and in most cases not more than 2 weight percent of the zeolite. The framework will normally include some alumina and the silica:alumina ratio will usually be at least 30:1, in the as-synthesized conditions of the zeolite. A preferred zeolite beta catalyst is made by steaming an initial boron-containing zeolite containing at least 1 weight percent boron (as B.sub.2 O.sub.3) to result in an ultimate alpha value no greater than 10 and preferably no greater than 5.

Detailed Description Text (32):
The zeolite will be composited with a matrix material to form the finished catalyst and for this purpose conventional non-acidic matrix materials such as alumina, silica-alumina and silica are suitable with preference given to silica as a non-acidic binder, although non-acidic aluminas such as alpha boehmite (alpha alumina monohydrate) may also be used, provided that they do not confer any substantial

degree of acidic activity on the matrixed catalyst. The use of silica as a binder is preferred since alumina, even if non-acidic in character, may tend to react with the zeolite under hydrothermal reaction conditions to enhance its acidity. The zeolite is usually composited with the matrix in amounts from 80:20 to 20:80 by weight, typically from 80:20 to 50:50 zeolite:matrix. Compositing may be done by conventional means including mulling the materials together followed by extrusion of pelletizing into the desired finished catalyst particles. A preferred method for extruding the zeolite with silica as a binder is disclosed in U.S. Pat. No. 4,582,815. If the catalyst is to be steamed in order to achieve the desired low acidity, it is performed after the catalyst has been formulated with the binder, as is conventional.

Detailed Description Text (53):
A slack wax with the properties shown in Table 3 above and containing 30 wt % oil based on bulk solvent dewaxing (35 wt % oil by ASTM D3235) was processed by hydrocracking over a 1.5 mm trilobe NiW/fluorided alumina catalyst of the type described in Table 4 above (4.8 wt. pct. Ni, 22.3 wt. pct. W). The catalyst was sulfided and fluorided in-situ using o-fluorotoluene at a level of 600 ppm fluorine for one week at a temperature of 725.degree. F. (385.degree. C.) before introducing the slack wax. The hydrocracking was carried out with fluorine maintenance at 25 ppm F using o-fluorotoluene under the following conditions:

Detailed Description Text (76):
A zeolite beta sample with a bulk SiO.sub.2 /Al.sub.2 O.sub.3 ratio of 40:1 was extruded with alumina to form a 65/35 weight percent cylindrical extrudate. This material was then dried, calcined and steamed to reduce the alpha to 55. Platinum was incorporated by means of ion exchange using Pt(NH.sub.3).sub.4 Cl.sub.2. The final Pt loading was 0.6 weight percent. This catalyst was then charged to the reactor and sulfided as described above. Hydrogen was fed to the reactor together with the same slack wax described in Example 3 in cocurrent downflow under the following conditions:

Detailed Description Paragraph Table (4):
TABLE 4 LHDC Catalyst Properties Form 1.5 mm cyl. 1.5 mm. tri. 1.5 mm. cyl. Pore Volume, cc/gm 0.331 0.453 0.426 Surface Area, m.sup.2 /gm 131 170 116 Nickel, wt. pct. 4.8 4.6 5.6 Tungsten, wt. pct. 22.3 23.8 17.25 Fluorine, wt. pct. -- -- 3.35 Silica, wt. pct. -- -- 2 Alumina, wt. pct. -- -- 60.3 Real Density, gm/cc 4.229 4.238 4.023 Particle Density, gm/cc 1.744 1.451 1.483 Packing Density, gm/cc 1.2 0.85 0.94

Detailed Description Paragraph Table (13):
TABLE 8 Example No. 3 NiW/ 7 8 Catalyst alumina 4.alpha. Pt/beta 55.alpha. Pt/beta Pressure, psig 2000 400 1750 400 2000 Lube yield, wt. pct 46 55-58 61 51 41 KV, 100.degree. F., cS 5.0 5.8 6.0 5.8 7.0 Lube VI 142 135-137 133-134 127 121

CLAIMS:

6. A process according to claim 1 in which the hydrocracking catalyst of step (a) comprises alumina as an acidic support material.

L7: Entry 7 of 8 File: USPT Jan 20, 1998

DOCUMENT-IDENTIFIER: US 5709852 A
TITLE: Ethylene oxide/propylene oxide/ethylene oxide (EO/PO/EO) triblock copolymer carrier blends

Brief Summary Text (5):
Further, personal care products, such as toothpastes, mouthwashes, cosmetic creams, gels and lotions, antiperspirants, deodorants, and over-the-counter medicaments such as salves and ointments, are subject to freeze-thaw cycles during shipment and storage. Subjecting personal care products to several freeze thaw cycles can alter the rheology of the product, creating a product dispensing problem when the product becomes too hard or too soft or too viscous, and hence, difficult to use.

Brief Summary Text (6):
The art has attempted to solve this stability problem in personal care products. To this end, the art has experimented with either EO/PO/EO solid or liquid triblock copolymers in personal care products. Specifically, WO 95/01155, discloses the use of a non-ionic surfactant as a stabilizing agent in antibody containing oral compositions at levels of 0.01-6% by weight of the oral composition. Preferred non-ionic surfactants are the solid EO/PO/EO triblock copolymers known as PLURONIC.RTM. F 68, F 88 and F 108. U.S. Pat. No. 5,374,368 describes the use of liquid EO/PO/EO triblock co-polymers (PLURONIC.RTM. L 31 and L 35) in stable hydrogen peroxide releasing dental care compositions at levels of 55-90% by weight of the dental care composition. U.S. Pat. No. 3,740,421 discloses gel forming solid EO/PO/EO triblock copolymers useful in cosmetic and personal care formulations at levels of approximately 20-25% by weight. Preferred solid EO/PO/EO triblock copolymers have a molecular weight of 4,600-16,000. Said solid EO/PO/EO triblock copolymers form a gel when added to an aqueous solution. U.S. Pat. No. 3,867,533 discloses aqueous gel compositions containing solid EO/PO/EO triblock copolymers, having a molecular weight of 6,450-20,000 useful at levels of approximately 20% by weight. Said compositions are useful in preparing cosmetic formulations. U.S. Pat. No. 3,997,458 discloses solid triblock co-polymers of EO/PO/EO useful in wound cleansing compositions at levels of approximately 10% by weight. Said EO/PO/EO copolymers have a molecular weight of 5,000-13,500 (e.g., PLURONIC.RTM. F 98, F 108--available from BASF Corporation, Mt. Olive, N.J.). U.S. Pat No. 4,382,078 discloses water based aerosol compositions containing a dimethylether propellant and solid EO/PO/EO triblock copolymers at levels of 1-6% by weight. U.S. Pat. No. 4,465,663 discloses clear aqueous cosmetic gels containing solid EO/BO(butylene oxide)/EO triblock copolymers at levels of approximately 20%. U.S. Pat. No. 5,035,880 discloses a stable dentrifice compositions containing a cetylpyridinium bactericide and EO/PO/EO solid triblock copolymers (PLURONIC.RTM. F 127), and polyethylene glycol at levels of 15-80% by weight. U.S. Pat. No. 4,476,107 discloses a mouthwash containing EO/BO(butylene oxide)/EO triblock copolymers at levels of 0.5-5.0% by weight. U.S. Pat. No. 5,057,307 discloses oral hygiene gels containing non-ionic surfactants, coating substances; and viscosifiers. Said non-ionic surfactants are PLURONIC.RTM. F 108 and F 127 available from BASF Corporation, Mt. Olive, N.J. U.S. Pat. No. 5,256,396 discloses a topical composition comprising an EO/PO/EO solid triblock copolymer (PLURONIC.RTM. F 127) used at a level of more than 10% to about 17% by weight. EPO-546-627A discloses mouthwash compositions comprising solid EO/PO/EO triblock copolymers such as PLURONIC.RTM. L 108, 88 at levels of 0.5-3% by weight. EP 0-551-626 discloses a thermoreversible pharmaceutical gel comprising

solid EO/PO/EO triblock copolymer such as PLURONIC.RTM. F 127 at a level of 10 to 30% by weight. U.S. Pat. No. 5,073,368 discloses mouthwashes containing solid EO/PO/EO triblock copolymers such as PLURONIC.RTM. F 87 at levels of 0.1-3% by weight. W 0 93/13750 discloses an ocular cleansing composition comprising solid PLURONIC.RTM. P 85 and paste PLURONIC.RTM. F 87 EO/PO/EO triblock copolymers. PLURONIC.RTM. P 85 is 4-9% by weight of the cleansing composition, PLURONIC.RTM. F 87 is 0.5-2% by weight of the cleansing composition. Finally, U.S. Pat. No. 5,096,698 discloses a dental creme composition containing a non-ionic triblock liquid EO/PO/EO copolymer or a solid triblock EO/PO/EO copolymer at levels of 0.1-5% by weight. Said copolymers help to prevent phase separation. PLURONIC.RTM. F 108 (solid) is most preferred, followed by PLURONIC.RTM. F 87, PLURONIC.RTM. F 127, and PLURONIC.RTM. L 72. Liquid and solid PLURONIC.RTM. surfactants are not used together in said dental creme composition.

Detailed Description Text (19):
Personal care formulations comprising these stable non-aqueous carrier blends may further contain other ingredients such as surfactants selected from anionic surfactants, such as sodium lauryl sulphate; sodium alkyl glyceryl ether sulfonate; alkyl benzene sulfonates. Further, small amounts of cationic surfactants,having a quaternary nitrogen, which show compatibility with the nonionic carrier blends of this invention can also be used. Various other materials may also be used in the formulating of personal care products. For example, in a dentrifice, dental abrasives consisting of freely divided silica, or calcium carbonate, calcium pyrophosphate, and hydrated alumina are added for polishing performance. Additionally, thickening agents such as xanthan gum, gum arabic, hydroxyethylcellulose can also be used to provide sufficient thickening consistency to the formulation. Also, flavoring agents such as peppermint, spearmint oils or preservatives, opacifying agents, buffer salts, sweeteners, anti-bacterial agents or anti-plaque agents, anti-inflammatory agents, anti-caries agents such as the fluoride salts can also be included in small amounts. Polymeric agents which accelerate the transport of active materials can also be included. Also, in cosmetic creams emollients such as glycerin, mineral oil and petrolatum can be added.

Detailed Description Text (20):
Personal care products are formulated according to methods known to those skilled in the art. Representative personal care product formulations are disclosed in: Cosmetics, Science and Technology, 2nd Edition, Vol. 1, Edited by M.S. Balsam, et al., and A Formulary of Cosmetic Preparations, Michael and Irene Ash, Chemical Publishing, N.Y., N.Y., both incorporated by reference herein.

L7: Entry 5 of 8 File: USPT Jul 6, 1999

DOCUMENT-IDENTIFIER: US 5919830 A
TITLE: Stable non-aqueous blends for personal care compositions

Brief Summary Text (5):
Further, personal care products, such as mouthwashes, cosmetic creams, gels and lotions, antiperspirants, deodorants, and over-the-counter medicaments such as salves and ointments, which are formulated comprising a non-aqueous blend, are subject to freeze-thaw cycles during shipment and storage. Subjecting such personal care products to several freeze thaw cycles can alter the rheology of the product, creating a product dispensing problem when the product becomes too hard or too soft or too viscous, and hence, difficult to use. Thus, there is a need for a non-aqueous carrier which offers a wide formulation flexibility and has the ability to withstand the extremes of transportation temperatures without significantly altering the rheology and the integrity of the composition.

Brief Summary Text (6):
The art is replete with non-aqueous carrier blend formulations. Specifically, WO 95/01155, discloses the use of a non-ionic surfactant as a stabilizing agent in antibody containing oral compositions at levels of 0.01-6% by weight of the oral composition. Preferred non-ionic surfactants are the solid EO/PO/EO triblock copolymers known as PLURONIC.RTM. F 68, F 88 and F 108. U.S. Pat. No. 5,374,368 describes the use of liquid EO/PO/EO triblock co-polymers (PLURONIC.RTM. L 31 and L 35) in stable hydrogen peroxide releasing dental care compositions at levels of 55-90% by weight of the dental care composition. U.S. Pat. No. 3,740,421 discloses gel forming solid EO/PO/EO triblock copolymers useful in cosmetic and personal care formulations at levels of approximately 20-25% by weight. Preferred solid EO/PO/EO triblock copolymers have a molecular weight of 4,600-16,000. Said solid EO/PO/EO triblock copolymers form a gel when added to an aqueous solution. U.S. Pat. No. 3,867,533 discloses aqueous gel compositions containing solid EO/PO/EO triblock copolymers, having a molecular weight of 6,450-20,000 useful at levels of approximately 20% by weight. Said compositions are useful in preparing cosmetic formulations. U.S. Pat. No. 3,997,458 discloses solid triblock co-polymers of EO/PO/EO useful in wound cleansing compositions at levels of approximately 10% by weight. Said EO/PO/EO copolymers have a molecular weight of 5,000-13,500 (e.g., PLURONIC.RTM. F 98, F 108 - available from BASF Corporation, Mt. Olive, N.J.). U.S. Pat. No. 4,382,078 discloses water based aerosol compositions containing a dimethylether propellant and solid EO/PO/EO triblock copolymers at levels of 1-6% by weight. U.S. Pat. No. 4,465,663 discloses clear aqueous cosmetic gels containing solid EO/BO(butylene oxide)/EO triblock copolymers at levels of approximately 20%. U.S. Pat. No. 5,035,880 discloses a stable dentrifice compositions containing a cetylpyridinium bactericide and EO/PO/EO solid triblock copolymers (PLURONIC.RTM. F 127), and polyethylene glycol at levels of 15-80% by weight. U.S. Pat. No. 4,476,107 discloses a mouthwash containing EO/BO(butylene oxide)/EO triblock copolymers at levels of 0.5-5.0% by weight. U.S. Pat. No. 5,057,307 discloses oral hygiene gels containing non-ionic surfactants, coating substances; and viscosifiers. Said non-ionic surfactants are PLURONIC.RTM. F 108 and F 127 available from BASF Corporation, Mt. Olive, N.J. U.S. Pat. No. 5,256,396 discloses a topical composition comprising an EO/PO/EO solid triblock copolymer (PLURONIC.RTM. F 127) used at a level of more than 10% to about 17% by weight. EPO-546-627A discloses mouthwash compositions comprising solid EO/PO/EO triblock

copolymers such as PLURONIC.RTM. 108, 88 at levels of 0.5-3% by weight. EP 0-551-626 discloses a thermoreversible pharmaceutical gel comprising solid EO/PO/EO triblock copolymer such as PLURONIC.RTM. F 127 at a level of 10 to 30% by weight. U.S. Pat. No. 5,073,368 discloses mouthwashes containing solid EO/PO/EO triblock copolymers such as PLURONIC.RTM. F 87 at levels of 0.1-3% by weight. WO 93113750 discloses an ocular cleansing composition comprising solid PLURONIC.RTM. P 85 and paste PLURONIC.RTM. F 87 EO/PO/EO triblock copolymers. PLURONIC.RTM. P 85 is 4-9% by weight of the cleansing composition, PLURONIC.RTM. F 87 is 0.5-2% by weight of the cleansing composition. U.S. Pat. No. 5,096,698 discloses a dental creme composition containing a non-ionic triblock liquid EO/PO/EO copolymer or a solid triblock EO/PO/EO copolymer at levels of 0.1-5% by weight. Said copolymers help to prevent phase separation. PLURONIC.RTM. F 108 (solid) is most preferred, followed by PLURONIC.RTM. F 87, PLURONIC.RTM. F 127, and PLURONIC.RTM. L 72. Liquid and solid PLURONIC.RTM. surfactants are not used together in said dental creme composition. Finally, U.S. Pat. No. 5,709,852 discloses a nonaqueous carrier for personal care compositions comprising: (a) nonionic liquid triblock EO/PO/EO copolymer and (b) nonionic solid triblock EO/PO/EO copolymer.

Detailed Description Text (6):
Personal care formulations comprising these stable non-aqueous carrier blends may further contain other ingredients such as surfactants selected from anionic surfactants, such as sodium lauryl sulphate; sodium alkyl glyceryl ether sulfonate; alkyl benzene sulfonates. Further, small amounts of cationic surfactants having a quaternary nitrogen, which show compatibility with the nonionic carrier blends of this invention can also be used. Various other materials may also be used in the formulating of personal care products. For example, in a dentrifice, dental abrasives consisting of finely divided silica, or calcium carbonate, calcium pyrophosphate, and hydrated alumina are added for polishing performance. Additionally, thickening agents such as xanthan gum, gum arabic, hydroxyethylcellulose can also be used to provide sufficient thickening consistency to the formulation. Also, flavoring agents such as peppermint, spearmint oils or preservatives, opacifying agents, buffer salts, sweeteners, anti-bacterial agents or anti-plaque agents, anti-inflammatory agents, anti-caries agents such as the fluoride salts can also be included in small amounts. Polymeric agents which accelerate the transport of active materials can also be included. Also, in cosmetic creams emollients such as glycerin, mineral oil and petrolatum can be added.

Detailed Description Text (7):
Personal care products are formulated according to methods known to those skilled in the art. Representative personal care product formulations are disclosed in: Cosmetics, Science and Technology, 2nd Edition, Vol. 1, Edited by M. S. Balsam, et al., and A Formulary of Cosmetic Preparations, Michael and Irene Ash, Chemical Publishing, N.Y., N.Y., both incorporated by reference herein.

L13: Entry 20 of 28 File: USPT Sep 28, 1999

DOCUMENT-IDENTIFIER: US 5958361 A
TITLE: Ultrafine metal oxide powders by flame spray pyrolysis

Brief Summary Text (5):
To date, use of such fine and superfine ceramic particle powders has been severely limited due to the cost of such powders. In general, intensive grinding (milling) of substances such as alumina or titania to produce fine powders has not been commercially successful, as the process is both time and energy intensive, and generally results in particles of non-regular shape and broad particle size distribution. It is very difficult to obtain ultra-fine powders simply by milling.

Brief Summary Text (6):
Several processes have been proposed to produce ultra-fine ceramic particles. Techniques can be roughly classified into three categories, two based on the phase in which processing occurs, i.e. the gas and liquid phases, the third being hybrid aerosol techniques.

Brief Summary Text (7):
Gas phase syntheses include metal vapor condensation and oxidation, sputtering, laser-ablation, plasma-assisted chemical vapor deposition, and laser-induced chemical vapor deposition. Liquid phase processing encompasses precipitation techniques, and sol-gel processing. Aerosol techniques include spray drying, spray pyrolysis, and flame oxidation/hydrolysis of halides.

Brief Summary Text (9):
Of the aerosol processing techniques available for production of ceramic powders, spray pyrolysis and flame oxidation of halides are the primary methods used to produce ultrafine powders. In both methods, submicron to micron sized droplets of solutions of metal salts or alkoxides are produced by standard aerosolization techniques. In spray pyrolysis, the resulting aerosol is thermolyzed (see FIG. 1), to pyrolytically convert the aerosol droplet to an individual ceramic particle of the same stoichiometry as the parent solution. Thermal events in the process include solvent evaporation, solute precipitation, thermal conversion of the precipitate to a ceramic, and sintering of the particle to full density.

Brief Summary Text (10):
Spray pyrolysis is most commonly used for the preparation of monometallic ceramic powders. The resultant powders typically have sizes in the 100-10,000 nm range and are frequently polycrystalline. Hollow particles and fragments of hollow particles are often produced if processing conditions are not carefully adjusted. Attempts to produce multimetallic oxide powders often lead to crystallites of different stoichiometry within each particle as a result of differences in the rates of precipitation of the metals, as salts or hydroxides, from solution. In addition, the particle sizes produced are controlled by the size of droplets within the aerosol and the weight percent dissolved solids in the solution. The final particle size decreases with smaller initial droplet sizes and lower concentrations of dissolved solids in solution. Unfortunately, precursor aerosolization rates, and thus production rates, generally decrease as droplet sizes decrease as one moves from mechanical atomization techniques, such as high velocity shear, to ultrasonic atomization and other more complex methods. A lower concentration of dissolved

solids also has the effect of decreasing production rates. Thus, spray pyrolysis can produce ultrafine particles only by sacrificing production rates. Furthermore, single crystal oxide and multimetallic oxide powders are very difficult to produce.

Brief Summary Text (11):
With reference to FIG. 1, during spray pyrolysis, an aerosol droplet 1 containing a precursor dissolved in solvent, is subject to evaporation 2 during which the aerosol droplet is reduced in size and converted to a heterogenous particle containing precipitated solids 3 on the exterior and a solution of precursor 4 on the interior. Further heating effects drying and decomposition 5 to form a solid particle 6 containing mixed amorphous oxides and hydroxides, which upon sintering 7 leads to fully dense polycrystalline particles 8 and/or porous or hollow polycrystalline particles 9.

Brief Summary Text (12):
Flame oxidation of halide aerosols is the most common technique for the large-scale production of ultrafine ceramic powders. An aerosol droplet of halide is oxidized/hydrolyzed in a flame to form a "ceramic vapor" of metal-oxide molecules. This "vapor" then condenses to form particles via a nucleation and growth mechanism coupled with significant particle coalescence. See, e.g., A. Gurav, T. Kodas, T. Pluym, Y. Xiong, "Aerosol Processing of Materials", AEROSOL SCIENCE AND TECHNOLOGY, 19 411-52 (1993). The flame oxidation process is similar to the gas-phase syntheses shown in FIG. 2. Both titania, used in the paint industry, and silica, used primarily for polymer reinforcement, are produced in multi-ton quantities by flame oxidation/hydrolysis of their respective chlorides, TiCl.sub.4 and SiCl.sub.4. Use of these techniques generate large quantities of corrosive and toxic HCl and in addition require toxic reagents and expensive highly reactive precursors.

Brief Summary Text (14):
To date, homogeneous ultrafine, multimetallic powders have not been produced using flame oxidation of metal halide aerosols. The literature suggests that different vapor pressures of the product metal oxides lead to different nucleation and growth rates of the respective oxides from the vapor phase, with the oxide having the lower vapor pressure condensing first to form solid particles. Subsequent condensation of the second metal oxide, both as monometallic particles and as a coating on the existing particles, leads to inhomogeneity both within each particle and between particles. A. Gurav et al., op.cit. Finally, there is a strong likelihood that there will soon be legislation/regulation of chlorine use in the environment that may limit future use of flame oxidation of halides as a route to ultrafine ceramic powders for technical use.

Brief Summary Text (16):
Still another approach to processing ultrafine powders relies on plasma and laser induced decomposition of volatile inorganic, organometallic and metalloorganic precursors in the gas phase (see FIG. 2). These methods are the most common techniques for processing ultrafine non-oxide powders, specifically nitrides and carbides. H. J. Lee et al. and K-H. Kim et al., op.cit. Typical syntheses involve passing a volatile precursor in a controlled atmosphere through a plasma, laser beam, or furnace, which provides the activation energy necessary to convert the precursor to a ceramic. Strutt et al. have synthesized silicon nitride powders by laser-driven reaction of various silane (e.g. SiH.sub.4 and Si.sub.2 H.sub.6) precursors and NH.sub.3. P. R. Strutt, K. E. Gonsalves, T. D. Xiao, "Synthesis of Polymerized Preceramic Nanoparticle Powders by Laser Irradiation of Metalorganic Precursors", NANOSTRUCTURED MATERIALS 1[1] 21-5 (1992). Silicon carbonitride powders can be produced via a plasma induced reaction of SiCl.sub.4 with CH.sub.4 and NH.sub.3. This processing approach is also equipment and energy intensive and presents difficulties in processing mixed metal powders. Furthermore, powder production rates are typically of the order of milligrams to a few grams per hour.

Brief Summary Text (19):
In U.S. Pat. No. 5,358,695 is disclosed a process for the manufacture of nanosize metal oxide and metal carbide ceramic particles by preparing particles containing the corresponding metal nitrate or acetate associated with a solid carbonaceous support, and introducing these particles into a high temperature, oxygen rich combustion zone, combusting the solid support and forming metal oxide powders by condensation of low vapor pressure ceramic species. Alternately, the solid fuel, typically a carbohydrate, is dissolved in water with the metal salt and sprayed into a heated zone that evaporates the water and then the resulting dry particles are combusted. In a continuous process, an aqueous solution of sucrose and zirconium or magnesium nitrate is dispersed into a low temperature (300-350.degree. C.) drying and carbonization zone, producing an aerosol of dry particles, following which the aerosol continues its passage into a high temperature oxidation zone maintained at c.a. 1250.degree. K. to 1500.degree. K.

Brief Summary Text (20):
The process of U.S. Pat. No. 5,358,695 has several drawbacks, however, First, an aerosol of dry, carbonaceous particles must first be produced. Additionally, these carbonaceous particles, containing metal nitrates, are potentially explosive, and if the residence time in the oxidizing atmosphere is insufficient or the temperature too low, the desired metal oxide particles may be contaminated with residual carbon. The yield of the process disclosed is also low, being in the range of only 0.1 to 0.2 grams per minute. Finally, since the ceramics are said to condense from the gas phase, heterogenous multi-metal oxide particles are expected within the particles, and stoichiometry is expected to vary between particles, as described by G. L. Messing et al., op.cit.

Detailed Description Text (15):
Examples of such preparations as heretofore as heretofore described may be found in U.S. Pat. Nos. 2,881,198; 3,455,980; 5,008,422; 5,099,052; 5,416,298; and other publications such as: C. L. Frye, "Pentacoordinate Silicon Derivatives IV. Alkylammonium Siliconate Salts Derived From 1,2-Diols", J. AMER. CHEM. SOC., 92:5, Mar. 11, 1970 pp. 1205 & 1210; C. L. Frye et al. "Pentacoordinate Silicon Compounds V.. Novel Silatrane Chemistry", J. AMER. CHEM. SOC., 93:25 Dec. 15, 1971 pp. 6805-6811; K. A. Blohowiak et al., "Synthesis of Penta-Alkoxn INORG. AND ORGANOMETALLIC POLYMERS WITH SPECIAL PROPERTIES and Penta-aryloxy Silicates Directly From SiO.sub.2 ", Kluwer Academic Publishers, Netherlands, 1992 pp. 99-111; R. M. Laine et al., "Synthesis of Pentacoordinate Silicon Complexes From SIO.sub.2 ", Nature, vol. 353, October 1991, pp. 642-644; K. W. Chew et al., "Inorganic Polymers Derived From Silica and Alumina, An Ion Conducting Polymer From the Reaction of BaSi (OCH.sub.2 CH.sub.2 O).sub.3 with Tetraethylene Glycol", which are herein incorporated by reference.

Detailed Description Text (29):
The glycolato polymetallooxane may be first prepared, isolated in solid form, and dissolved in the solvent, generally after being finely ground, or alternatively, the desired solvent or solvent mixture may be added to the glycolato polymetallooxane immediately following synthesis. In addition to the glycolato polymetallooxane and solvent or mixture of solvents, other additives may be incorporated into the precursor solution in minor amounts. Examples include soluble metal compounds, i.e. in the form of soluble oxides, hydroxides, nitrates, acetates, etc., desired to adjust overall stoichiometry and/or incorporate trace metals in the final product for electrical, ferromagnetic, or piezoelectric properties, or for imparting color; viscosity adjusting agents to raise or lower the viscosity of the precursor solution, and surfactants to promote aerosolization or adjust aerosol particle size. Such additions are generally present in most minor amounts, i.e., less than 5 weight percent, and preferably less than 1 weight percent based on precursor solution weight.

Detailed Description Text (30):

The size of powder particles depends upon the aerosol droplet size as well as the precursor solution glycolato polymetallooxane concentration. It is preferable to maintain glycolato polymetallooxane concentration in the range of 5-15 weight percent.

Detailed Description Text (31):
Aerosolization may be accomplished by several well known technologies. For example, the precursor solution may be atomized by flow through a restrictive nozzle at high pressure, or by flow into a high volume, low pressure gas stream. When such atomizers are used, the high volume gas stream should be air, air enriched with oxygen, or preferably substantially pure oxygen. When high pressure atomization through a restrictive orifice is used, the orifice is preferably surrounded by jets of one of the above gases, preferably oxygen. More than one atomizer for aerosolization may be positioned within the flame pyrolysis chamber. Other aerosol-producing methods such as those described in the foregoing references, for example ultrasonic or piezoelectric droplet formation, may be used. However, some of these techniques may undesirably affect production rate. Ultrasonic generation is preferred, the aerosol generator generating ultrasound through reasonant action of the oxygen flow and the liquid in a chamber.

Detailed Description Text (32):
The aerosol is ignited by suitable means, for example laser energy, glow wire, electrical discharge, but is preferably ignited by means of an oxyhydrogen or hydrocarbon gas/oxygen torch. Prior to initiating combustion, the flame pyrolysis chamber is preheated to the desired operating range of 500.degree. C. to 2000.degree. C., preferably 700.degree. C. to 1500.degree. C., and most preferably 800.degree. C. to 1200.degree. C. Preheating improves particle size distribution and minimizes water condensation in the system. Preheating may be accomplished through the use of the ignition torch alone, by feeding and combusting pure solvent, i.e. ethanol, through the atomizer, by resistance heating or containment in a muffle furnace, combinations of these methods, or other means.

Detailed Description Text (33):
The flame pyrolysis apparatus as described herein, in conjunction with the volatile flammable organic solvent, creates a very steep temperature gradient that allows the oxide species generated by combustion to coalesce but limits the rate such that the resulting nanoparticles do not neck. The presence of water vapor in the product stream due to the combustion of solvent and organic ligands of the glycolato polymetallooxane further serves to passivate the particle surfaces, also contributing to a low degree of necking. The ultrafine particles are frequently single crystals of the desired metal oxide or mixed metal oxide.

Detailed Description Text (38):
Example 1, Alumina Precursor Synthesis

Detailed Description Text (41):
Al(OH).sub.3.xH.sub.2 O was purchased from Chattem Chemical, Chattanooga, Tenn.) and MgO from Aldrich Chem. Co. One kilogram of Al(OH).sub.3.xH.sub.2 O (equivalent to 5.6 mol as Al.sub.2 O.sub.3 after pyrolysis at 900.degree. C., determined by loss on ignition) and 220 g of MgO.xH.sub.2 O (5.6 mol as MgO) were added to a 5 L flask with 2.2 L of triethanolamine (TEA, 16.8 mol) and 2 L of ethylene glycol. The solution was heated to distill off byproduct water and ethylene glycol, resulting in approximately 3 L of clear solution after 4-6 hours with approximately 400 ml of H.sub.2 O and 1.8 L of ethylene glycol collected as distillate. This solution can be used directly for flame spray pyrolysis, or the remaining solvent can be removed and the resulting solid, which is pure TEAHMg(TEA-Al).sub.2 can be stored for later use. Note that TEA is used in cosmetics and is not considered a hazardous or polluting material. Ethylene glycol is a major component of antifreeze. The product is designated as SP1.

Detailed Description Text (48):
The flame spray pyrolysis system shown in the drawings consists of an aerosol generator with fluid feed and reservoir; a cylindrical quartz precursor ignition chamber 7.5 cm in diameter and 15 cm in length; a 5.0 cm diameter quartz pipe ending in a tee; four concentric wire-in-cylinder electrostatic precipitators (ESPs), 5.0 cm in diameter and 1 m in length, connected in parallel-series; and exhaust piping flow control valves located downstream of the collection zone.

Detailed Description Text (49):
One embodiment of a flame spray pyrolysis vessel is illustrated in FIG. 3. At 301 is the flame spray pyrolysis chamber, which is constructed of temperature-resistant material such as quartz, VYCORO.RTM. glass, ceramic, or if externally cooled, of metal. At 303 is a multichannel ceramic torch, for example of alumina, fed with oxygen or oxygen enriched gas through supply tube 305, and combustible gas, preferably hydrogen or natural gas through supply tube 307. Ceramic precursor solution is supplied through valve 309 from precursor solution supply line 311 which leads to the precursor solution tank 313. Valve 309 may be switched to an off position or to the solvent supply line 315 for purposes of preheating the chamber or during change or replenishment of the precursor solution supply tank.

Detailed Description Text (50):
From valve 309 the precursor solution or solvent flows through precursor delivery tube 317 which terminates just beyond the outlet end of oxygen delivery tube 319, forming an aerosol in the area 321 which is ignited by torch 303 to form a flame front which advances toward the opposite end of chamber 301 and forms, through decomposition and oxidation of the glycolato polymetallooxane contained in the precursor solution, a cloud of nucleating and coalescing ceramic vapor 323 which, by the time the exit tube 327 of the apparatus is reached, has formed a cloud 325 of nanosized ceramic particles which flow out the exit 329 to the collection apparatus.

Detailed Description Text (51):
The precursor is introduced into the ignition chamber using twin high-shear (Bernoulli) aerosol generators with high velocity oxygen as the atomizing gas. The aerosol generator consists of a 0.4 mm (i.d.) precursor delivery tube oriented orthogonally to the oxygen flow from a 0.8 mm (i.d.) tube (FIG. 3b). Twin aerosol generators were used to maintain high throughput and stabilize the flame. Ignition at each aerosol generator is accomplished using a natural gas/oxygen pilot torch. Torch tip temperatures can exceed 2000.degree. C. The torch was fabricated from a 15 mm (o.d.) multichannel alumina tube to avoid metallic contamination of the powder which may occur using a stainless steel torch. System temperatures are monitored by Pt-Pt 13% Rh thermocouples (TA instruments, New Castle, Del.) inserted directly into the product stream, 50-100 cm downstream from the ignition chamber (FIG. 3a); the upper use temperature of the thermocouples does not allow placement in the combustion zone.

Detailed Description Text (54):
The flame spray pyrolysis system, described above, is effective at handling throughputs greater than 1000 ml of solution containing 5-10 weight % solids per hour. Much higher rates are anticipated to be possible with simple increases in the dimensions of the reactor and a higher throughput aerosol generator. The precursor (glycoloato polymetallooxane) viscosity is lowered by ethanol addition, which also serves as an energy source for the combustion reaction. The precursor is introduced into a combustion chamber via an aerosol generator, shown in FIG. 3, where the individual droplets are combusted rapidly, resulting in the production of crystalline, homogeneous, ultrafine powder.

Detailed Description Text (56):
Prior to precursor flame spray pyrolysis, the system is preheated using the ignition torches to avoid condensing byproduct water in the system. Pure ethanol is

fed through the aerosol generators to finish preheating the combustion chamber to operating temperatures (800-1200.degree. C.). When the system reaches equilibrium, approximately 5 minutes, the aerosol generator feed is switched to the precursor. Precursor flow rate is controlled by head pressure generated by a compressed nitrogen tank as well as the precursor solution viscosity. No direct correlation between head pressure and precursor flow rate was determined. Substitution of a pump or other means of metered precursor may be desirable.

Detailed Description Text (60):
Powder X-Ray Diffraction (XRD). The flame spray pyrolysis product powders were analyzed by powder x-ray diffraction using a Rigaku 2.theta. Double Crystal Wide Angle Goniometer (Tokyo, Japan). Specimens (0.1-0.2 g) were packed in a glass specimen holder and placed in the diffractometer. Full scans were measured from 5-80.degree. 2.theta. at a scan speed of 5.degree. 2.theta./min in 0.05.degree. 2.theta. increments using Cu (K.theta.) radiation operated at 40 kV and 100 Amps. The Debye-Scherrer crystallite sizes determined from peak broadening were obtained at a scan speed of 0.5.degree. 2.theta./min in 0.01.degree. 2.theta. increments. Peak positions were compared with standard JCPDS files, 21-1152 and 16-394 for spinel and .delta.-alumina, respectively, to identify crystalline phases.

Detailed Description Text (64):
Powder Characterization. The ultrafine spinel and alumina powders produced using the flame spray pyrolysis process are found to be dense, crystalline and spherical, as detailed below. The particles range in size primarily from 10-100 nm. with some much larger particles seen in the SEM micrographs. Reaction conditions and results are summarized in Table I. FIGS. 6a and 7a are TEM and SEM photomicrographs of powders formed from the SP1 precursor, while FIGS. 6b and 7b are TEM and SEM photomicrographs of SP2 derived powders. FIG. 8 contains the X-ray diffraction spectrum of these powders, while FIGS. 9a and 9b are the TEM photomicrographs, respectively, of titania powder prepared from TP1 precursor and TiN powder prepared by ammonolysis as set forth below.

Other Reference Publication (8):
A. Gurav, T. Kodas, T. Pluym, Y. Xiong, "Aerosol Processing of Materials", Aerosol Science and Technology, 19, 411-52 (1993), no month.

Other Reference Publication (16):
P.R. Strutt, K.E. Gonsalves, T.D. Xiao, "Synthesis of Polymerized Preceramic Nanoparticle Powders by Laser Irradiation of Metalorganic Precursors." Nanostructured Materials 1 [1] 21-5 (1992), no month.

Other Reference Publication (22):
"Processing of Silicon Carbide-Mullite-Alumina Nanocomposites", Yoshio Sakka, J.Am.Ceram.Soc. 78 [2], 479-86 (1995), no month.

Other Reference Publication (24):
"Preparation of Monodisperse, Spherical Alumina Powders from Alkoxides", J. Am. Ceram. Soc. 74 [9] 2263-69 (1991), no month.

Other Reference Publication (25):
"Synthesis of Submicrometer Mullite Powder via High-Temperature Aerosol Decomposition", K. A. Moore, et al, J. Am. Ceram. Soc. 75 [1], 213-15 (1992), no month.

Other Reference Publication (30):
"Texture Evolution of SnO.sub.2 Synthesized by Pyrolysis of an Aerosol", M. Vallet-Regi, et al, J. Mater. Res., vol. 8, No. 1, Jan. 1993.

Other Reference Publication (35):
"Preparation of Small Particle Stabilized Zirconia by Aerosol Pyrolysis" A.R.

Pebler, J. Mater. Res., vol. 5, No. 4, pp. 680-682, Apr. 1990.

CLAIMS:

1. A process for the preparation of ultrafine metal oxide or mixed metal oxide ceramic particles having mean particle sizes in the range of 2-500 nm, said process comprising:

a) aerosolizing a ceramic precursor solution comprising one or more glycolato polymetallooxanes in a volatile organic solvent or volatile flammable organic solvent mixture, said glycolato polymetallooxane(s) present in said volatile solvent(s) in an amount of from 1 weight percent to 30 weight percent of said ceramic precursor solution, said glycolato polymetallooxane(s) having bound therein in excess of 90% of the metal content of said metal oxide or mixed metal oxide, to form a ceramic precursor solution aerosol containing droplets of said ceramic precursor solution;

b) supplying oxygen to said ceramic precursor solution aerosol in an amount at least stoichiometrically equivalent to combustible material contained in said ceramic precursor solution aerosol to form an aerosol and oxygen mixture;

c) igniting said aerosol an oxygen mixture and forming metal oxide or mixed metal oxide ceramic particles thereby and a gaseous effluent containing products of combustion; and

d) separating said metal oxide or said mixed metal oxide ceramic particles from said gaseous effluent to recover an ultrafine ceramic particle product.

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L13: Entry 27 of 28 File: JPAB May 22, 2002

JP00 02

DOCUMENT-IDENTIFIER: JP 2002145735 A (JP 2002145735 A)

TITLE: COMPOSITION PACKAGED IN AEROSOL DEVICE, COMPRISING ALUMINA NANOPARTICLE

Abstract Text (1):
PROBLEM TO BE SOLVED: To provide a cosmetic composition packaged in an aerosol device and comprising alumina nanoparticles.

Abstract Text (2):
SOLUTION: Provided are a composition packaged in an aerosol device comprising a liquid phase and a propellant. The liquid phase comprises, in a cosmetically acceptable medium, nanoparticles comprising at least 10% by weight of alumina and exhibiting a number-average primary size of between 2 to 200 nm. The propellant is chosen from dimethyl ether, 3-5C alkanes, 1,1- difluoroethane, mixtures of dimethyl ether and of 3-5C alkanes or mixtures of 1,1-difluoroethane and of dimethyl ether and/or of 3-5C alkanes, a process for the cosmetic treatment of the hair; and use as styling product.

How can I get this?

L13: Entry 23 of 28 File: USPT Jan 6, 1998

DOCUMENT-IDENTIFIER: US 5705194 A
TITLE: Pharmaceutical compositions containing polyalkylene block copolymers which gel at physiological temperature

Abstract Text (1):
There is disclosed a pharmaceutical composition which gels at physiological temperature. The composition is comprised of a block copolymer containing one or more polyoxyethylene blocks and one or more polyoxy (higher alkylene) blocks wherein at least some of the blocks are linked together by a linking group characterised in that the linking group is an oxymethylene group, and a therapeutic agent. The therapeutic agent is present as (i) nanoparticles of the therapeutic agent having the block copolymer adsorbed on the surface thereof, (ii) a suspension in a solution of the block copolymer, or (iii) as an aqueous solution in a solution of the block copolymer.

Brief Summary Text (7):
U.S. Pat. No. 4,534,959, there is described a composition in an aerosol container. When sprayed the composition gels on the surface of living tissue. This composition contains a polyoxyethylene-polyoxypropylene copolymer. However the copolymer must be present in high percentage. Further, the therapeutic agent must be solubilized. U.S. Pat. No. 3,867,533 relates to another aqueous gel composition; U.S. Pat. No. 4,465,663 is similar and relates to cosmetic gels.

Brief Summary Text (12):
(I) nanoparticles of the therapeutic agent having the block copolymer adsorbed on the surface thereof,

Detailed Description Text (42):
Suitable therapeutic or diagnostic agents useful in the three types of formulations used in the present invention (i.e., nanoparticles, solutions and suspensions) be selected from a variety of known classes of therapeutic or diagnostic agents including, for example, analgesics, anti-inflammatory agents, anthelmintics, anti-arrhythmic agents, antibiotics (including penicillins), anticoagulants, antidepressants, antidiabetic agents, antiepileptics, antihistamines, antihypertensive agents, antimuscarinic agents, antimycobacterial agents, antineoplastic agents, immunosuppressants, antithyroid agents, antiviral agents, anxiolytic sedatives (hypnotics and neuroleptics), astringents, beta-adrenoceptor blocking agents, blood products and substitutes, cardiac inotropic agents, contrast media, corticosteroids, cough suppressants (expectorants and mucolytics), diagnostic agents, diagnostic imaging agents, diuretics, dopaminergics (antiparkinsonian agents), haemostatics, immuriological agents, lipid regulating agents, muscle relaxants, parasympathomimetics, parathyroid calcitonin and biphosphonates, prostaglandins, radio- pharmaceuticals, sex hormones (including steroids), anti-allergic agents, stimulants and anoretics, sympathomimetics, thyroid agents, vasodilators and xanthines. Preferred therapeutic or diagnostic agents include those intended for oral administration, topically, ophalmic and nasal application. A description of these classes of therapeutic agents and diagnostic agents and a listing of species within each class can be found in Martindale, The Extra Pharmacopoeia, Twenty-ninth Edition, The Pharmaceutical Press, London, 1989. The therapeutic or diagnostic agents are commercially

available and/or can be prepared by techniques known in the art.

Detailed Description Text (49):
The present block copolymers are also particularly useful with therapeutic agents in solutions and suspensions for oral application such as the following: A- oral solutions--acetaminophen, aminobenzoate, aminophylline, amprolium, aspirin, carphenzaine maleate, chloramphenicol, clindamycin palmirate hydrochloride, cloxacillin sodium, cloxacillin sodium, cyanocobalamin Co, cyclosprine, dihydrotachysterol, diphenoxylate hydrochloride, atropine sulfate, doxepine hydrochloride, ergocalciferol, ergoloid mesylates, fluphenzaine hydrochloride, glycerin, guaifenesin, theophyline, haloperidol, hyoscyamine sulfate, isosorbide, levocarnitine, mesoridazine besylate, methadone hydrochloride, methenamine mandelate, metoclopramide, nafcillin sodium, neomycin sulfate, nortriptyline hydrochloride, oxacillin sodium, oxycocone hydrochloride, paramethadione, penicillin G potassium, perphenazine, prednisone, prochlorperazine edisulate, promazine hydrochloride, theophylline, guaifenesin, thioridazine hydrochloride, thiothixene hydrochloride, trimethadione, vancomycin hydrochloride and any of their combinations B- for oral suspensions--acetopminophen, alumina, magnesia, simethicone, amoxicillin, amphotericin B, ampicillin, bacampicillinhydrochloride, bephenium hydroxynaphthoate, cefaclor, cefadroxil, cephalexin, cephradine, chloramphenicol palmitate, chlorothiazide, chlorprothixene, chlestyramine, clavulanate potassium, colestipol hydrochloride, colistin sulfate, cyclacillin, demeclocycline, diazoxide, dicloxacillin sodium, doxycycline, doxycycline calcium, erythromycin estolate, erythromycin ethylsuccinate, erythromycin stearate, erythromycin ethylsuccinate, furazolidone, griseofulvin, hetacillin, hydrocortisone cypionate, hydroxyzine pamoate, ipodate calcium, levopropoxyphene napsylate, mgaldrate, magnesium carbonate, magnesium trisilicate, meprobamtate, methacycline hydrochloride, methamine mandelate, methyldopa, minocycline hydrochloride, nalidixic acid, nitrofurantoin, novobiocin calcium, nystatin, oxytetracycline calcium, penicillin G benzathine, penicillin V, penicillin V benzathine, phenytoin, primidone, probenecid, propoxyphene napsylate, psyllium hydrophilic mucilloid, pyrantel pamoate, pyrvinium pamoate, sulfamethizole, sulfamethoxazole, sulfisoxazole acetyl, tetracycline, thiabendazole, thioridazine, triflupromazine, trimethoprim, trisulfapyrimidines, troleandomycin and any of their combinations.

Detailed Description Text (50):
Nanoparticles

Detailed Description Text (51):
The present invention also has utility to administer and stablize nanoparticles wherein the copolymer is absorbed on the surface of the nanoparticles.

Detailed Description Text (52):
Nanoparticles, described in U.S. Pat. No. 5,145,684, are particles consisting of a poorly soluble therapeutic or diagnostic agent onto which are adsorbed a non-crosslinked surface modifier, and which have an average particle size of less than about 400 nanometers (nm). These nanoparticles provide for increased bioavailability and for improved diagnostic characteristics compared to other materials having larger sizes.

Detailed Description Text (53):
The nanoparticles can comprise a wide variety of therapeutic or diagnostic agents. (Therapeutic agents are sometimes referred to as drugs or pharmaceuticals. The diagnostic agent referred to is typically a contrast agent such as an x-ray contrast agent but can also be other diagnostic materials.) The therapeutic or diagnostic agent exists as a discrete, crystalline phase. The crystalline phase differs from a non-crystalline or amorphous phase which results from precipitation techniques, such as described in EPO 275,796.

Detailed Description Text (55):

The described block copolymers can be used with NSAIDS. Surface modified nanoparticles comprising an NSAID, e.g., naproxen, demonstrate reduced gastric irritation and/or a more rapid onset of action following oral administration.

Detailed Description Text (59):
The described nanoparticles can be prepared in accordance with methods disclosed in the prior including U.S. Pat. No. 5,145,684 which is incorporated by reference herein in. The method of the reference comprises the steps of dispersing a therapeutic or diagnostic agent in a liquid dispersion medium and applying mechanical means in the presence of grinding media to reduce the particle size of the therapeutic or diagnostic agent to an effective average particle size of less than about 400 nm. The particles can be reduced in size in the presence of the copolymer. Alternatively, the particles can be contacted with the copolymer surface modifier after attrition.

Detailed Description Text (64):
Preparation Conditions for Nanoparticles

CLAIMS:

1. A pharmaceutical composition comprised of

a) a triblock copolymer having the following repeating units in random order

--((E.sub.r P.sub.s E.sub.r)CH.sub.2 O).sub.z --

wherein

P is oxypropylene,

E is oxyethylene,

r is an integer from 2 to 160,

s is an integer from 15 to 65, and

z is an integer from 2 to 50, wherein the triblock copolymer gels at physiological temperature, and

b) a therapeutic agent, wherein the therapeutic agent is present as

(I) nanoparticles of the therapeutic agent having the block copolymer adsorbed on the surface thereof,

(II) a suspension in a solution of the block copolymer, or

(III) an aqueous solution in a solution of the block copolymer.

L7: Entry 6 of 8 File: USPT Oct 27, 1998

DOCUMENT-IDENTIFIER: US 5827508 A
**** See image for Certificate of Correction ****
TITLE: Stable photoprotective compositions

Brief Summary Text (4):
The damaging effects of sunlight on skin are well documented. Contrary to what most people believe, it is not necessary that one sunbathe to suffer the ill-effects of excessive UV exposure. In fact, significant damage can be done just by routine day to day activities in sunlight. The major short term hazard of prolonged exposure to sunlight is erythema, i.e. sunburn. In addition to the short term hazard are long term hazards such as malignant changes in the skin surface. Numerous epidemiologic studies demonstrate a strong relationship between sunlight exposure and human skin cancer. Another long term hazard of ultraviolet radiation is premature aging of the skin. This condition is characterized by wrinkling and pigment changes of the skin, along with other physical changes such as cracking, telangiectasis, solar dermatoses, ecchymoses, and loss of elasticity. The adverse effects associated with exposure to UV radiation are more fully discussed in DeSimone, "Sunscreen and Suntan Products," Handbook of Nonprescription Drugs, 7th Ed., Chapter 26, pp. 499-511 (American Pharmaceutical Association, Washington, D.C.; 1982); Grove and Forbes, "A Method for Evaluating the Photoprotection Action of Sunscreen Agents Against UV-A Radiation," International Journal of Cosmetic Science, 4, pp. 15-24 (1982); and U.S. Pat. No. 4,387,089, DePolo, issued Jun. 7, 1983; all of these references being incorporated by reference herein in their entirety. Hence, although the immediate effects of ultraviolet radiation may be cosmetically and socially gratifying, the long term hazards are cumulative and potentially serious.

Brief Summary Text (5):
The fact that these effects are taken seriously by the general public is suggested by considering the sun protection product market. This market has grown considerably in recent years and many new products are introduced each year. What used to be looked upon as a seasonal business is no longer seen as such. Sunscreen agents are now included in a diversity of personal care products, particularly cosmetic type products which are worn on a daily basis.

Brief Summary Text (42):
The sunscreen 4,4'-methoxy-t-butyldibenzoylmethane, which is also known as butyl methoxydibenozylmethane, is commercially available under the trademark Parsol.RTM. 1789 from Givaudan Corporation. See CTFA International Cosmetic Ingredient Dictionary, fifth edition, 1993, p. 79, which is incorporated by reference herein in its entirety. The sunscreen 4-isopropyldibenzoylmethane, which is also known as isopropyl dibenzoylmethane, is commercially available under the trademark Eusolex.RTM. 8020 from Merck. See CTFA International Cosmetic Ingredient Dictionary, fifth edition, 1993, pp. 347-348, which is incorporated by reference herein in its entirety.

Brief Summary Text (46):
Zinc oxide can be represented by the chemical formula ZnO. It is generally a white or yellowish-white odorless powder. See The Merck Index, Tenth Edition, 1983, p. 1457, entry 9952, and International Cosmetic Ingredient Dictionary, Fifth Edition, 1993, pp. 788-789, both references being incorporated by reference herein in their

entirety.

Brief Summary Text (49):
The surface treatment materials used to surface treat the zinc oxide particles of the present invention can comprise from about 0.1% to about 50%, more preferably from about 0.25% to about 25%, and most preferably from about 0.5% to about 10% by weight of the surface-treated zinc oxide. Nonlimiting classes of surface treatment materials useful for treating the zinc oxide particles include silicones, fatty acids, proteins, peptides, amino acids, N-acyl amino acids, monoglycerides, diglycerides, triglycerides, mineral oils, silica, phospholipids, sterols, hydrocarbons, polyacrylates, alumina and mixtures thereof. Preferred herein is silicone-treated zinc oxide.

Brief Summary Text (64):
An example of alumina useful as a surface treatment material is boehmite.

Brief Summary Text (70):
The topical carrier can be in a wide variety of forms. For example, emulsion carriers, including, but not limited to, oil-in-water, water-in-oil, water-in-oil-in-water, and oil-in-water-in-silicone emulsions, are useful herein. These emulsions can cover a broad range of viscosities, e.g., from about 100 cps to about 200,000 cps. These emulsions can also be delivered in the form of sprays using either mechanical pump containers or pressurized aerosol containers using conventional propellants. These carriers can also be delivered in the form of a mousse. Other suitable topical carriers include aqueous-based single phase solvents, e.g., water, alcohols, glycols, polyols, and the like. Examples of topical carrier systems useful in the present invention are described in the following references, an of which are incorporated herein by reference in their entirety: "Sun Products Formulary," Cosmetics & Toiletries, vol. 105, pp. 122-139 (December 1990); "Sun Products Formulary," Cosmetics & Toiletries, vol. 102, pp. 117-136 (March 1987); U.S. Pat. No. 4,960,764 to Figueroa et al., issued Oct. 2, 1990; U.S. Pat. No. 4,254,105 to Fukuda et al., issued Mar. 3, 1981; U.S. Pat. No. 4,976,953, to Orr et al., issued Dec. 11, 1990; and U.S. Pat. No. 5,073,372, to Turner et al., issued Dec. 17, 1991.

Brief Summary Text (71):
Other forms of topical carriers are also useful. When the sunscreening composition is an aerosol spray or mousse, the carrier can also utilize any of the conventional propellants to deliver the material as a foam (in the case of a mousse) or as a fine, uniform spray (in the case of an aerosol). Examples of suitable propellants include materials such as trichlorofluoromethane, dichlorodifluoromethane, difluoroethane, dimethylether, propane, n-butane or isobutane. A more complete disclosure of propellants useful herein can be found in Sagarin, Cosmetics Science and Technology, 2nd Edition, Vol. 2, pp. 443-465 (1972), which is incorporated herein by reference in its entirety.

Brief Summary Text (72):
Suitable spray containers are well known in the art and include conventional, non-aerosol pump sprays, i.e., "atomizers," aerosol containers or cans having propellant, as described above, and also pump aerosol containers utilizing compressed air as the propellant. Pump aerosol containers are disclosed, for example, in U.S. Pat. Nos. 4,077,441, Mar. 7, 1978, Olofsson and 4,850,577, Jul. 25, 1989, both incorporated by reference herein, and also in U.S. Ser. No. 07/839,648, Gosselin, Lund, Sojka, and Lefebvre, filed Feb. 21, 1992, "Consumer Product Package Incorporating A Spray Device Utilizing Large Diameter Bubbles." Pump aerosols hair sprays using compressed air are also currently marketed by The Procter & Gamble Company under their tradename VIDAL SASSOON AIRSPRAY hair sprays.

Brief Summary Text (80):
Dihydroxyacetone, which is also known as DHA or 1,3-dihydroxy-2-propanone, is a

white to off-white, crystalline powder. This material can be represented by the chemical formula C.sub.3 H.sub.6 O.sub.3 and the following chemical structure. ##STR5## The compound can exist as a mixture of monomers and dimers, with the dimers predominating in the solid crystalline state. Upon heating or melting, the dimers break down to yield the monomers. This conversion of the dimeric form to the monomeric form also occurs in aqueous solution. Dihydroxyacetone is also known to be more stable at acidic pH values. See The Merck Index, Tenth Edition, entry 3167, p. 463 (1983), and "Dihydroxyacetone for Cosmetics", E. Merck Technical Bulletin, 03-304 110, 319 897, 180 588; both of these references being incorporated herein by reference in their entirety.

Brief Summary Text (107):
The compositions of the present invention can comprise a wide range of other optional components. These additional components should be pharmaceutically acceptable. The CTFA Cosmetic Ingredient Handbook, Second Edition, 1992, which is incorporated by reference herein in its entirety, describes a wide variety of nonlimiting cosmetic and pharmaceutical ingredients commonly used in the skin care industry, which are suitable for use in the compositions of the present invention. Nonlimiting examples of functional classes of ingredients are described at page 537 of this reference. Examples of these functional classes include: abrasives, absorbents, anti-acne agents, anticaking agents, antifoaming agents, antimicrobial agents, antioxidants, binders, biological additives, buffering agents, bulking agents, chelating agents, chemical additives, colorants, cosmetic astringents, cosmetic biocides, denaturants, drug astringents, external analgesics, film formers, fragrance components, humectants, opacifying agents, pH adjusters, preservatives, propellants, reducing agents, skin bleaching agents, skin-conditioning agents (humectants, miscellaneous, and occlusive).

Brief Summary Text (108):
Some nonlimiting examples of these additional components cited in the CTFA Cosmetic Ingredient Handbook as well as other materials useful herein, include the following: vitamins and derivatives thereof (e.g. tocopherol, tocopherol acetate, retinoic acid, retinol, retinoids, and the like); polymers for aiding the film-forming properties and substantivity of the composition (such as a copolymer of eicosene and vinyl pyrrolidone, an example of which is available from GAF Chemical Corporation as Ganex.RTM. V-220); preservatives for maintaining the antimicrobial integrity of the compositions; other anti-acne medicaments (e.g., resorcinol, sulfur, salicylic acid, erythromycin, zinc, and the like); skin bleaching (or lightening) agents including but not limited to hydroquinone, kojic acid; antioxidants; chelators and sequestrants; skin treating agents such as alpha-hydroxy acids such as lactic acid and glycolic acid, and aesthetic components such as fragrances, pigments, colorings, essential oils, skin sensates, astringents, skin soothing agents, skin healing agents and the like, nonlimiting examples of these aesthetic components include panthenol and derivatives (e.g. ethyl panthenol), aloe vera, pantothenic acid and its derivatives, clove oil, menthol, camphor, eucalyptus oil, eugenol, menthyl lace, witch hazel distillate, allantoin, bisabolol, and dipotassium glycyrrhizinate.

Other Reference Publication (3):
International Cosmetic Ingredient Dictionary, Fifth Edition, 1993, pp. 788-789.

CLAIMS:

4. A composition according to claim 3 wherein said surface-treated zinc oxide is surface treated with a material selected from the group consisting of silicones, fatty acids, proteins, peptides, amino acids, N-acyl amino acids, monoglycerides, diglycerides, triglycerides, mineral oils, silica, phospholipids, sterols, hydrocarbons, polyacrylates, alumina, and mix thereof.

L23: Entry 2 of 3 File: USPT Oct 27, 1998

DOCUMENT-IDENTIFIER: US 5827508 A
** See image for Certificate of Correction **
TITLE: Stable photoprotective compositions

Brief Summary Text (49):
The surface treatment materials used to surface treat the zinc oxide particles of the present invention can comprise from about 0.1% to about 50%, more preferably from about 0.25% to about 25%, and most preferably from about 0.5% to about 10% by weight of the surface-treated zinc oxide. Nonlimiting classes of surface treatment materials useful for treating the zinc oxide particles include silicones, fatty acids, proteins, peptides, amino acids, N-acyl amino acids, monoglycerides, diglycerides, triglycerides, mineral oils, silica, phospholipids, sterols, hydrocarbons, polyacrylates, alumina and mixtures thereof. Preferred herein is silicone-treated zinc oxide.

Brief Summary Text (64):
An example of alumina useful as a surface treatment material is boehmite.

Brief Summary Text (70):
The topical carrier can be in a wide variety of forms. For example, emulsion carriers, including, but not limited to, oil-in-water, water-in-oil, water-in-oil-in-water, and oil-in-water-in-silicone emulsions, are useful herein. These emulsions can cover a broad range of viscosities, e.g., from about 100 cps to about 200,000 cps. These emulsions can also be delivered in the form of sprays using either mechanical pump containers or pressurized aerosol containers using conventional propellants. These carriers can also be delivered in the form of a mousse. Other suitable topical carriers include aqueous-based single phase solvents, e.g., water, alcohols, glycols, polyols, and the like. Examples of topical carrier systems useful in the present invention are described in the following references, an of which are incorporated herein by reference in their entirety: "Sun Products Formulary," Cosmetics & Toiletries, vol. 105, pp. 122-139 (December 1990); "Sun Products Formulary," Cosmetics & Toiletries, vol. 102, pp. 117-136 (March 1987); U.S. Pat. No. 4,960,764 to Figueroa et al., issued Oct. 2, 1990; U.S. Pat. No. 4,254,105 to Fukuda et al., issued Mar. 3, 1981; U.S. Pat. No. 4,976,953, to Orr et al., issued Dec. 11, 1990; and U.S. Pat. No. 5,073,372, to Turner et al., issued Dec. 17, 1991.

Brief Summary Text (71):
Other forms of topical carriers are also useful. When the sunscreening composition is an aerosol spray or mousse, the carrier can also utilize any of the conventional propellants to deliver the material as a foam (in the case of a mousse) or as a fine, uniform spray (in the case of an aerosol). Examples of suitable propellants include materials such as trichlorofluoromethane, dichlorodifluoromethane, difluoroethane, dimethylether, propane, n-butane or isobutane. A more complete disclosure of propellants useful herein can be found in Sagarin, Cosmetics Science and Technology, 2nd Edition, Vol. 2, pp. 443-465 (1972), which is incorporated herein by reference in its entirety.

Brief Summary Text (72):
Suitable spray containers are well known in the art and include conventional, non-

aerosol pump sprays, i.e., "atomizers," aerosol containers or cans having propellant, as described above, and also pump aerosol containers utilizing compressed air as the propellant. Pump aerosol containers are disclosed, for example, in U.S. Pat. Nos. 4,077,441, Mar. 7, 1978, Olofsson and 4,850,577, Jul. 25, 1989, both incorporated by reference herein, and also in U.S. Ser. No. 07/839,648, Gosselin, Lund, Sojka, and Lefebvre, filed Feb. 21, 1992, "Consumer Product Package Incorporating A Spray Device Utilizing Large Diameter Bubbles." Pump aerosols hair sprays using compressed air are also currently marketed by The Procter & Gamble Company under their tradename VIDAL SASSOON AIRSPRAY hair sprays.

Brief Summary Text (107):
The compositions of the present invention can comprise a wide range of other optional components. These additional components should be pharmaceutically acceptable. The CTFA Cosmetic Ingredient Handbook, Second Edition, 1992, which is incorporated by reference herein in its entirety, describes a wide variety of nonlimiting cosmetic and pharmaceutical ingredients commonly used in the skin care industry, which are suitable for use in the compositions of the present invention. Nonlimiting examples of functional classes of ingredients are described at page 537 of this reference. Examples of these functional classes include: abrasives, absorbents, anti-acne agents, anticaking agents, antifoaming agents, antimicrobial agents, antioxidants, binders, biological additives, buffering agents, bulking agents, chelating agents, chemical additives, colorants, cosmetic astringents, cosmetic biocides, denaturants, drug astringents, external analgesics, film formers, fragrance components, humectants, opacifying agents, pH adjusters, preservatives, propellants, reducing agents, skin bleaching agents, skin-conditioning agents (humectants, miscellaneous, and occlusive).

CLAIMS:

4. A composition according to claim 3 wherein said surface-treated zinc oxide is surface treated with a material selected from the group consisting of silicones, fatty acids, proteins, peptides, amino acids, N-acyl amino acids, monoglycerides, diglycerides, triglycerides, mineral oils, silica, phospholipids, sterols, hydrocarbons, polyacrylates, alumina, and mix thereof.

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L13: Entry 28 of 28 File: DWPI Mar 22, 2002

DERWENT-ACC-NO: 2002-428900
DERWENT-WEEK: 200249

TITLE: Aerosol composition for use as a hair dressing, comprises specified nanoparticles containing alumina in a cosmetically acceptable medium and propellant

Basic Abstract Text (1):
NOVELTY - A composition packaged in an aerosol comprising a liquid phase comprising 2-200 nm nanoparticles containing at least 10 wt.% of alumina in a cosmetically acceptable medium and dimethylether, a 3-5C alkane, 1,1-difluoroethane, a mixture of dimethylether and 3-5C alkane or 1,1-difluoroethane and dimethylether and/or 3-5C alkane as a propellant.

Equivalent Abstract Text (1):
NOVELTY - A composition packaged in an aerosol comprising a liquid phase comprising 2-200 nm nanoparticles containing at least 10 wt.% of alumina in a cosmetically acceptable medium and dimethylether, a 3-5C alkane, 1,1-difluoroethane, a mixture of dimethylether and 3-5C alkane or 1,1-difluoroethane and dimethylether and/or 3-5C alkane as a propellant.

Standard Title Terms (1):
AEROSOL COMPOSITION HAIR DRESS COMPRISE SPECIFIED CONTAIN ALUMINA COSMETIC ACCEPT MEDIUM PROPELLANT

| 12 cosmetics

| 14 nanoparticles

L13: Entry 21 of 28 File: USPT Aug 4, 1998

DOCUMENT-IDENTIFIER: US 5788973 A
TITLE: Photobluing-resistant cosmetic/dermatological compositions comprising TiO.sub.2 pigments and benzotriazole silicones

Abstract Text (1):
Topically applicable, photobluing-resistant cosmetic/dermatological compositions, well suited for improved photoprotection of human skin and/or the scalp against the damaging effects of UV-A and UV-B irradiation and/or for the therapeutic treatment of a disease state of the skin or scalp, comprise (a) an effective photoprotective amount of at least one titanium dioxide (nano) pigment and (b) an effective photobluing-reducing amount of at least one benzotriazole silicone.

Brief Summary Text (3):
The present invention relates to novel photobluing-resistant cosmetic/dermatological compositions containing photoprotective amounts of titanium dioxide pigments and at least one specific benzotriazole silicone.

Brief Summary Text (11):
For the purpose of limiting this photoreactivity phenomenon, surface-treated TiO.sub.2 pigments have been described. Thus, EP-B-0,461,130 describes TiO.sub.2 nanoparticles treated with phosphate anions. Likewise, cosmetics containing TiO.sub.2 pigments surface-treated with silica or alumina are known. However, these treatments are expensive and difficult to carry out.

Brief Summary Text (18):
More particularly according to the present invention, at least one benzotriazole silicone as defined above is formulated into cosmetic and/or dermatological compositions containing photoprotective amounts of titanium dioxide pigments for decreasing the photobluing due to the presence of said pigments in said compositions.

Brief Summary Text (19):
In one specific embodiment of this invention, cosmetic and/or dermatological compositions are provided, in particular for the photoprotection of the skin and/or hair, comprising, in a cosmetically acceptable vehicle, diluent or carrier, titanium dioxide pigments and at least one benzotriazole silicone as defined above, with the proviso that the subject compositions advantageously do not contain either benzene-1,4-di(3-methylidene-10-comphorsulfonic acid) or 4,4',-methoxy-t-butyldibenzoylmethane.

Brief Summary Text (21):
Example 2 of FR-A-2,695,560, also assigned to the assignee hereof, describes a screening cosmetic composition comprising titanium dioxide nanopigments, a benzotriazole silicone in accordance with those of the present invention and 4,4'-methoxy-t-butyldibenzoylmethane. According to the invention of said application, the goal is to stabilize 4,4',-methoxy-t-butyldibenzoylmethane using a screening polymer of the benzatriazole silicone type. However, no reduction in the photobluing of titanium dioxide pigments via by the presence of any benzotriazole silicone is described or suggested.

Brief Summary Text (23):
The present invention also features a cosmetic treatment regimen for protecting the skin and/or hair against ultraviolet radiation, in particular solar radiation, comprising topically applying thereto an effective amount of at least one composition as described above.

Brief Summary Text (48):
One of the essential characteristics of the compositions of the present invention is that they contain at least one titanium dioxide pigment. The pigments of this invention are known titanium dioxide pigments commonly used in the cosmetics field as fillers or as screening agents, which can be treated or untreated. Such pigments include titanium dioxide nanopigments. By "nanopigments" are intended pigments in which the average size of the elementary particles ranges from 5 to 100 nm.

Brief Summary Text (50):
The treated pigments may, for example, be treated with alumina, silica, aluminum compounds, silicon compounds, sodium compounds, iron oxides, iron esters, stearic acid or glycerol.

Brief Summary Text (52):
(i) silica and alumina, such as the products "Microtitanium dioxide MT 500 SA" and "Microtitanium dioxide MT 100 SA" marketed by Tayca, and the products "Tioveil Fin", "Tioveil OP", "Tioveil MOTG" and "Tioveil IPM" marketed by Tioxide,

Brief Summary Text (53):
(ii) alumina and aluminum stearate, such as the product "Microtitanium dioxide MT 100 T" marketed by Tayca,

Brief Summary Text (54):
(iii) alumina and aluminum laurate, such as the product "Microtitanium dioxide MT 100 S" marketed by Tayca,

Brief Summary Text (56):
(v) silica, alumina and silicone, such as the products "Microtitanium dioxide MT 100 SAS", "Microtitanium dioxide MT 600 SAS" and "Microtitanium dioxide MT 500 SAS" marketed by Tayca,

Brief Summary Text (59):
(viii) alumina and stearic acid, such as the product "UVT-M160" marketed by Remira,

Brief Summary Text (60):
(ix) alumina and glycerol, such as the product "UVT-M212" marketed by Remira,

Brief Summary Text (61):
(x) alumina and silicone, such as the product "UVT-M262" marketed by Remira.

Brief Summary Text (64):
Other constituents advantageously comprising the compositions of the invention include, in particular, oils, waxy compounds, thickeners, emulsifiers or gelling agents, and are those which are conventionally employed in the cosmetics and/or dermatological fields.

Brief Summary Text (72):
The compositions of the present invention can also contain various ingredients conventionally used in the cosmetics, dermatological or dermopharmaceutical fields, such as coloring materials, solvents (water, alcohols and the like), preservatives, fragrances, moisturizing active principles, agents which absorb or block ultraviolet radiation (organic sunscreening agents or inorganic pigments other than titanium dioxides, in particular cerium and/or zinc oxides), pulverulent agents,

bactericidal agents and/or odor absorbers.

Brief Summary Text (73):
These compositions can, in addition, contain one or more hydrophilic and preferably lipophilic cosmetic or dermatological active agents or principles, in particular for treating and/or preventing skin conditions, such as acne, mycoses, eczema, rosacea, seborrhoeic dermatitides, solar dermatitides or cutaneous aging, and conditions of the scalp. These compositions are intended for topical skin treatment.

Brief Summary Text (76):
The subject compositions can be formulated, in particular, as simple or complex (O/W, W/O, O/W/O or W/O/W) emulsions, such as a cream, a milk, a lotion, a gel or a cream gel, as a powder or as a solid stick and can optionally be packaged as an aerosol and be provided in the form of a foam or spray.

CLAIMS:

1. A topically applicable, photobluing-resistant cosmetic/dermatological composition, comprising (a) an effective photoprotective amount of at least one titanium dioxide pigment and (b) an effective photobluing-reducing amount of at least one benzotriazole silicone having one of the following formulae (1) and (2): ##STR17## in which the radicals R, which may be identical or different, are each a C.sub.1 -C.sub.10 alkyl, phenyl, 3,3,3-trifluoropropyl or trimethylsilyloxy radical, at least 80% by number of the radicals R being methyl radicals; r is an integer ranging from 0 to 50, inclusive; s is an integer of ranging from 1 and 20, inclusive; u is an integer of ranging from 1 and 6, inclusive; t is an integer of ranging from 0 and 10, inclusive, with the proviso that t+u is equal to or greater than 3; and A is a monovalent radical bonded directly to a silicon atom which has the following formula (3): ##STR18## wherein the radicals Y, which may be identical or different, are each a C.sub.1 -C.sub.8 alkyl radical, a halogen atom or a C.sub.1 -C.sub.4 alkoxy radical, with the proviso that, in the latter event, two adjacent Y radicals on the same aromatic nucleus can together form an alkylidenedioxy radical in which the alkylidene moiety contains from 1 to 2 carbon atoms; X represents O or NH; Z represents hydrogen or a C.sub.1 -C.sub.4 alkyl radical; n is an integer ranging from 0 to 3, inclusive; m is 0 or 1; and p is an integer ranging from 1 to 10, inclusive, wherein such composition is devoid of the compounds benzene-1,4-di(3-methylidene-10-camphorsulfonic acid) and 4,4'-methoxy-t-butyldibenzoylmethane.

2. A topically applicable, photobluing-resistant cosmetic/dermatological composition, consisting essentially of (a) an effective photoprotective amount of at least one titanium dioxide pigment and (b) an effective photobluing-reducing amount of at least one benzotriazole silicone having one of the following formulae (1) and (2): ##STR19## in which the radicals R, which may be identical or different, are each a C.sub.1 -C.sub.10 alkyl, phenyl, 3,3,3-trifluoropropyl or trimethylsilyloxy radical, at least 80% by number of the radicals R being methyl radicals; r is an integer ranging from 0 to 50, inclusive; s is an integer of ranging from 1 and 20, inclusive; u is an integer of ranging from 1 and 6, inclusive; t is an integer of ranging from 0 and 10, inclusive, with the proviso that t+u is equal to or greater than 3; and A is a monovalent radical bonded directly to a silicon atom which has the following formula (3): ##STR20## wherein the radicals Y, which may be identical or different, are each a C.sub.1 -C.sub.8 alkyl radical, a halogen atom or a C.sub.1 -C.sub.4 alkoxy radical, with the proviso that, in the latter event, two adjacent Y radicals on the same aromatic nucleus can together form an alkylidenedioxy radical in which the alkylidene moiety contains from 1 to 2 carbon atoms; X represents O or NH; Z represents hydrogen or a C.sub.1 -C.sub.4 alkyl radical; n is an integer ranging from 0 to 3, inclusive; m is 0 or 1; and p is an integer ranging from 1 to 10, inclusive, wherein such composition is devoid of the compounds benzene-1,4-di(3-methylidene-10-

camphorsulfonic acid) and 4,4',-methoxy-t-butyldibenzoylmethane.

3. A method for treating a disease state of the skin or scalp, comprising topically applying thereto a therapeutically effective amount of a cosmetic/dermatological composition comprising (a) an effective photoprotective amount of at least one titanium dioxide pigment and (b) an effective photobluing-reducing amount of at least one benzotriazole silicone having one of the following formulae (1) and (2): ##STR21## in which the radicals R, which may be identical or different, are each a C.sub.1 -C.sub.10 alkyl, phenyl, 3,3,3-trifluoropropyl or trimethylsilyloxy radical, at least 80% by number of the radicals R being methyl radicals; r is an integer ranging from 0 to 50, inclusive; s is an integer of ranging from 1 and 20, inclusive; u is an integer of ranging from 1 and 6, inclusive; t is an integer of ranging from 0 and 10, inclusive, with the proviso that t+u is equal to or greater than 3; and A is a monovalent radical bonded directly to a silicon atom which has the following formula (3): ##STR22## wherein the radicals Y, which may be identical or different, are each a C.sub.1 -C.sub.8 alkyl radical, a halogen atom or a C.sub.1 -C.sub.4 alkoxy radical, with the proviso that, in the latter event, two adjacent Y radicals on the same aromatic nucleus can together form an alkylidenedioxy radical in which the alkylidene moiety contains from 1 to 2 carbon atoms; X represents O or NH; Z represents hydrogen or a C.sub.1 -C.sub.4 alkyl radical; n is an integer ranging from 0 to 3, inclusive; m is 0 or 1; and p is an integer ranging from 1 to 10, inclusive.

4. The cosmetic/dermatological composition as defined by claim 1, said at least one benzotriazole silicone having the formula (1).

5. The cosmetic/dermatological composition as defined by claim 1, said at least one benzotriazole silicone having the formula (2).

6. The cosmetic/dermatological composition as defined by claim 4, wherein formula (1) at least one of the following conditions is satisfied:

R is an alkyl radical;

r ranges from 0 to 15, inclusive;

s ranges from 1 to 10, inclusive;

n is other than zero;

Z is hydrogen or methyl;

m is 0, or m is 1 and X is O; and

p is 1.

7. The cosmetic/dermatological composition as defined by claim 6, wherein formula (1) n is 1 and Y is methyl, tert-butyl or a C.sub.1 -C.sub.4 alkoxy radical.

8. The cosmetic/dermatological composition as defined by claim 6, wherein formula (1) each of said conditions is satisfied.

9. The cosmetic/dermatological composition as defined by claim 4, said at least one benzotriazole silicone having the following formula (4): ##STR23## in which 0.ltoreq.r.ltoreq.10; 1.ltoreq.s.ltoreq.10; and D is the divalent radical: ##STR24##

10. The cosmetic/dermatological composition as defined by claim 9, wherein formula (4) r is 0, s is 1 and D is ##STR25##

11. The cosmetic/dermatological composition as defined by claim 9, wherein formula (4) r is 5, s is 5 and D is ##STR26##

12. The cosmetic/dermatological composition as defined by claim 1, comprising from 0.1% to 15% by weight of said at least one benzotriazole silicone.

13. The cosmetic/dermatological composition as defined by claim 12, comprising from 2% to 10% by weight of said at least one benzotriazole silicone.

14. The cosmetic/dermatological composition as defined by claim 1, said at least one titanium dioxide pigment having an average elementary particle size ranging from 5 to 100 nm.

15. The cosmetic/dermatological composition as defined by claim 1, comprising from 0.1% to 30% by weight of said at least one titanium dioxide pigment.

16. The cosmetic/dermatological composition as defined by claim 15, comprising from 0.2% to 25% by weight of said at least one titanium dioxide pigment.

17. The cosmetic/dermatological composition as defined by claim 1, formulated into a cosmetically/dermatologically acceptable, topically applicable vehicle, diluent or carrier therefor.

18. The cosmetic/dermatological composition as defined by claim 17, comprising an oil-in-water or water-in-oil emulsion.

19. The cosmetic/dermatological composition as defined by claim 17, comprising a cream, a milk, a lotion, a gel, a cream gel, a powder, a solid stick, a foam, or a spray.

20. The cosmetic/dermatological composition as defined by claim 1, further comprising at least one oil, wax, thickener, emulsifier and/or gelling agent.

21. The cosmetic/dermatological composition as defined by claim 1, further comprising at least one colorant, solvent, preservative, fragrance, moisturizer, UV screen other than TiO.sub.2, pulverulent, bactericide and/or odor absorber.

22. The cosmetic/dermatological composition as defined by claim 1, further comprising an effective amount of at least one bioactive agent for treating a disease state of the skin or scalp.

23. A treatment for protecting human skin and/or the scalp against the deleterious effects of ultraviolet irradiation, comprising topically applying thereto an effective photoprotecting amount of the cosmetic/dermatological composition as defined by claim 1.

24. A method for treating a disease state of the skin or scalp, comprising topically applying thereto a therapeutically effective amount of the cosmetic/dermatological composition as defined by claim 22.

26. A cosmetic/dermatological composition comprising (a) an effective photoprotective amount of at least one titanium dioxide pigment and (b) an effective photobluing-reducing amount of at least one benzotriazole silicone having one of the following formulae (1) and (2): ##STR27## in which the radicals R, which may be identical or different, are each a C.sub.1 -C.sub.10 alkyl, phenyl, 3,3,3-trifluoropropyl or trimethylsilyloxy radical, at least 80% by number of the radicals R being methyl radicals; r is an integer ranging from 0 to 50, inclusive; s is an integer of ranging from 1 and 20, inclusive; u is an integer of ranging from 1 and 6, inclusive; t is an integer of ranging from 0 and 10, inclusive, with the proviso that t+u is equal to or greater than 3; and A is a monovalent radical

bonded directly to a silicon atom which has the following formula (3): ##STR28## wherein the radicals Y, which may be identical or different, are each a C.sub.1 -C.sub.8 alkyl radical, a halogen atom or a C.sub.1 -C.sub.4 alkoxy radical, with the proviso that, in the latter event, two adjacent Y radicals on the same aromatic nucleus can together form an alkylidenedioxy radical in which the alkylidene moiety contains from 1 to 2 carbon atoms; X represents O or NH; Z represents hydrogen or a C.sub.1 -C.sub.4 alkyl radical; n is an integer ranging from 0 to 3, inclusive; m is 0 or 1; and p is an integer ranging from 1 to 10, inclusive, further comprising an effective amount of at least one bioactive agent for treating a disease state of the skin or scalp.

27. A treatment for protecting human skin and/or the scalp against the deleterious effects of ultraviolet irradiation, comprising topically applying thereto an effective photo-protecting amount of a cosmetic/dermatological composition, comprising (a) an effective photoprotective amount of at least one titanium dioxide pigment and (b) an effective photobluing-reducing amount of at least one benzotriazole silicone having one of the following formulae (1) and (2): ##STR29## in which the radicals R, which may be identical or different, are each a C.sub.1 -C.sub.10 alkyl, phenyl, 3,3,3-trifluoropropyl or trimethylsilyloxy radical, at least 80% by number of the radicals R being methyl radicals; r is an integer ranging from 0 to 50, inclusive; s is an integer of ranging from 1 and 20, inclusive; u is an integer of ranging from 1 and 6, inclusive; t is an integer of ranging from 0 and 10, inclusive, with the proviso that t+u is equal to or greater than 3; and A is a monovalent radical bonded directly to a silicon atom which has the following formula (3): ##STR30## wherein the radicals Y, which may be identical or different, are each a C.sub.1 -C.sub.8 alkyl radical, a halogen atom or a C.sub.1 -C.sub.4 alkoxy radical, with the proviso that, in the latter event, two adjacent Y radicals on the same aromatic nucleus can together form an alkylidenedioxy radical in which the alkylidene moiety contains from 1 to 2 carbon atoms; X represents O or NH; Z represents hydrogen or a C.sub.1 -C.sub.4 alkyl radical; n is an integer ranging from 0 to 3, inclusive; m is 0 or 1; and p is an integer ranging from 1 to 10, inclusive.

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DOCUMENT-IDENTIFIER: US 6376679 B2
TITLE: Photoprotective/cosmetic compositions comprising novel benz-x-azole-substituted silane/siloxane sunscreens

Abstract Text (1):
Photoprotective, topically applicable sunscreen/cosmetic compositions well suited for the UV-photoprotection of human skin and/or hair, comprise an effective UV-photoprotecting amount of at least one novel benz-x-azole-substituted silane or siloxane having the formula (1) or (2): ##STR1## A--SiR'.sub.1 R'.sub.2 R'.sub.3 (2)

Brief Summary Text (3):
The invention concerns new silicon-bearing derivatives of benz-x-azole that are liposoluble, photostable, and have an excellent absorption power in the UV radiation spectrum. The invention also pertains to compositions, particularly cosmetic compositions, containing these new derivatives, which can be used for photoprotection of the skin and/or hair against UV radiation, particularly solar radiation.

Brief Summary Text (6):
It is also a known fact that UV-A radiation, having a wavelength of between 320 and 400 nm, which causes the skin to tan, is likely to induce an alteration of the skin, particularly in the case of sensitive skin or skin that is continually exposed to solar radiation. In particular, UV-A rays cause the skin to lose its elasticity and wrinkles to appear, leading to premature skin aging. They promote the triggering of the erythematic reaction or amplify this reaction in certain subjects, and can even be the cause of phototoxic or photoallergic reactions. Consequently, for esthetic and cosmetic reasons, such as maintaining the natural elasticity of the skin, for example, more and more people wish to control the effect of UV-A radiation on their skin. It is therefore desirable to filter out UV-A radiation as well.

Brief Summary Text (9):
In addition to their filtration power, these anti-UV compounds must also have good cosmetic properties in the compositions containing them, good solubility in the usual solvents, especially fatty substances such as oils and greases, as well as good resistance to water and perspiration (persistence).

Brief Summary Text (13):
More specifically, according to this invention it was found that by grafting one or more benz-x-azole groups to a silicone chain, it was possible to arrive at new compounds that, in addition to excellent filtering properties in the UV-A and/or UV-B radiation range, provide very good solubility in the commonly-used organic solvents and particularly fatty substances such as oils, as well as excellent cosmetic properties, making them particularly appropriate for use as solar radiation filters in, or for the preparation of, cosmetic compositions intended for the protection of the skin and/or hair against ultraviolet radiation.

Brief Summary Text (43):
The compounds of the invention display excellent liposolubility and can thus be

used in large concentrations, thus providing the final compositions with very high protection indices; furthermore, they spread uniformly throughout conventional cosmetic media containing at least one fatty phase or a cosmetically acceptable organic solvent, and can thus be applied to the skin or the hair in order to act as an effective protective film.

Brief Summary Text (92):
The object of this invention is also a composition comprising a compound per formula (1) through (4) of the invention in an appropriate medium. The medium can be, for instance, a plastic material composition. It may also be appropriate for topical application. In this case the composition of the invention is a cosmetic composition which comprises a cosmetically acceptable medium.

Brief Summary Text (97):
The compositions according to the invention may furthermore contain pigments or nanopigments (average size of primary particles: generally between 5 nm and 100 nm, preferably between 10 nm and 50 nm) of metal oxides, either coated or not, such as nanopigments of oxides of titanium (amorphous or crystallized in rutile and/or octahedrite form), iron, zinc, zirconium, or cerium which are all well-known UV photoprotective agents in their own right. Furthermore, conventional coating agents consist of alumina and/or aluminum stearate. Such nanopigments of metal oxides, either coated or not, are described in patent applications EP-A-0518772 and EP-A-0518773.

Brief Summary Text (98):
The composition may contain the cosmetic additives commonly used in the field of cosmetics, such as fatty substances, organic solvents, silicones, thickeners, softeners, additional sun filters, anti-foaming agents, hydrating agents, scents, preservatives, surface-active agents, fillers, complexing agents, anionic, cationic, non-ionic, or amphoteric polymers or their mixtures, propellants, alkalinizing or acidifying agents, dyes, pigments or nanopigments, particularly those intended to provide an additional photoprotective effect through physical blocking of the ultraviolet radiation, or any other ingredient generally used in cosmetics, especially for the manufacture of sunscreen compositions.

Brief Summary Text (102):
The cosmetic composition of the invention can be used as a human epidermis or hair protection composition against ultraviolet rays, as a sunscreen composition, or as a make-up product.

Brief Summary Text (103):
The composition may occur in the form of a lotion, a thickened lotion, a gel, a creme, a milk, a powder, a solid stick, and may possibly be packaged as an aerosol in the form of a foam or spray.

Brief Summary Text (104):
When the cosmetic composition of the invention is specifically intended to protect the human epidermis against UV rays or as a sunscreen composition, it may occur in the form of a suspension or dispersion in solvents or fatty substances, or in the form of an emulsion (particularly an O/W or W/O emulsion, but preferably O/W) such as a creme or a milk, a vesicular dispersion, in the form of an ointment, gel, solid stick, or aerosol foam. The emulsions may furthermore contain anionic, non-ionic, cationic, or amphoteric surface-active agents.

Brief Summary Text (105):
When the cosmetic composition according to the invention is used to protect the hair, it can occur in the form of a shampoo, lotion, gel, or rinse composition to be applied before or after shampooing, before or after dyeing, before, during, or after a permanent or straightening treatment, a styling or treatment lotion or gel, a brushing or setting lotion or gel, hair spray, a permanent or straightening

composition or a hair dyeing or bleaching composition.

Brief Summary Text (106):
When the cosmetic composition according to the invention is specifically intended for depigmentation of the skin, it may occur in all the galenical forms normally used for topical application, particularly in the form of an aqueous, hydroalcohol, or oily solution, an oil-in-water or water-in-oil emulsion or multiple emulsion, an aqueous or oily gel, a liquid, pasty, or solid anhydrous product, a dispersion of oil in an aqueous phase using spherules, with the spherules possibly being polymeric nanoparticles such as nanospheres and nanocapsules or, better still, ionic and/or non-ionic lipid vesicles.

Brief Summary Text (107):
The composition may be more or less fluid and have the appearance of a white or colored creme, an ointment, a milk, a lotion, a serum, a paste, or a foam. It could possibly be applied to the skin in an aerosol form. It can also occur in a solid form and, for instance, in the form of a stick.

Brief Summary Text (108):
When the cosmetic composition according to the invention is used as an eyelash, eyebrow, skin or hair make-up product, such as a skin treatment creme, foundation, lipstick, eye shadow, rouge, eye liner, mascara, or coloring gel, it can occur in a solid or pasty, anhydrous or aqueous form, such as oil-in-water or water-in-oil emulsions, suspensions, or gels.

Brief Summary Text (111):
Finally, the object of the invention is a cosmetic process for protecting the skin and/or hair against ultraviolet radiation, particularly solar radiation, which involves the application of an effective quantity of the cosmetic composition defined above, or a compound per formula (1), (2), (3), or (4) as defined earlier, to the skin or hair.

Detailed Description Text (32):
Here we give a concrete example of a cosmetic composition according to the invention, that is, a sunscreen O/W emulsion:

CLAIMS:

22. A photostable sunscreen or cosmetic composition suited for the UV-photoprotection of human skin, hair or both, comprising an effective UV-photoprotecting amount of at least one benz-x-azole-substituted silane or siloxane having the formula (1) or (2): ##STR34## A--SiR'.sub.1 R'.sub.2 R'.sub.3 (2)

in which R is a C.sub.1 -C.sub.30 saturated or unsaturated hydrocarbyl radical, a C.sub.1 -C.sub.8 halogenated hydrocarbyl radical, or a trimethylsilyloxy radical; a is 1 or 2; the radicals R'.sub.1, R'.sub.2 and R'.sub.3, which may be identical or different, are each a C.sub.1 -C.sub.8 linear or branched alkyl or alkenyl radical; and A is a radical having the structural formula (I): ##STR35##

in which L is a divalent radical bonding said radical A to the silicon atom; the radicals R.sub.1 and R.sub.2, which may be identical or different, are each a hydrogen atom, a C.sub.1 -C.sub.10 linear or branched alkyl radical, a C.sub.2 -C.sub.8 linear or branched alkenyl radical, with the proviso that two adjacent R.sub.1 or R.sub.2 radicals may together form an alkylidenedioxy radical wherein the alkylidene moiety has 1 or 2 carbon atoms; Y is C or N; and X is O, NR.sub.3, S when Y is C, or C when Y is N, wherein R.sub.3 is a hydrogen atom, or a C.sub.1 -C.sub.8 alkyl radical; and n and m are, independently, 0 or 1, formulated into a topically applicable, cosmetically acceptable medium therefor.

23. The sunscreen or cosmetic composition as defined by claim 22, comprising from

0.1% to 20% by weight of said at least one benz-x-azole-substituted silane or siloxane (1) or (2).

24. The sunscreen or cosmetic composition as defined by claim 23, comprising from 0.5% to 10% by weight of said at least one benz-x-azole-substituted silane or siloxane (1) or (2).

25. The sunscreen or cosmetic composition as defined by claim 22, comprising an oil-in-water emulsion.

26. The sunscreen or cosmetic composition as defined by claim 22, comprising a water-in-oil emulsion.

27. The sunscreen or cosmetic composition as defined by claim 22, further comprising at least one additional hydrophilic or lipophilic organic UV-A and/or UV-B sunscreen.

28. The sunscreen or cosmetic composition as defined by claim 27, further comprising at least one cinnamic sunscreen, salicylic sunscreen, camphor sunscreen, triazine sunscreen, benzophenone sunscreen, dibenzoylmethane sunscreen, .beta.,.beta.-diphenyl-acrylate sunscreen, p-aminobenzoic acid sunscreen, sunscreen polymer or sunscreen silicone.

29. The sunscreen or cosmetic composition as defined by claim 22, further comprising a UV-photoprotecting effective amount of particulates of at least one inorganic pigment or nanopigment.

30. The sunscreen or cosmetic composition as defined by claim 29, said at least one pigment or nanopigment comprising titanium dioxide, zinc oxide, iron oxide, zirconium oxide, cerium oxide, or mixture thereof.

31. The sunscreen or cosmetic composition as defined by claim 22, further comprising at least one active agent for the artificial tanning or darkening of human skin, or for the artificial tanning and darkening of human skin.

32. The sunscreen or cosmetic composition as defined by claim 22, further comprising at least one cosmetically acceptable adjuvant or additive.

33. The sunscreen or cosmetic composition as defined by claim 32, said at least one adjuvant or additive comprising a fat, organic solvent, thickening agent, softener, silicone, anti-foaming agent, hydrating agent, fragrance, preservative, surfactant, filler, complexing agent, polymer, propellant, basifying or acidifying agent, dye, pigment, or mixture thereof.

34. The sunscreen or cosmetic composition as defined by claim 22, comprising a vesicular dispersion, lotion, cream, milk, gel, ointment, suspension, paste, dispersion, powder, solid stick, foam or spray.

35. The sunscreen or cosmetic composition as defined by claim 22, comprising a makeup.

36. The sunscreen or cosmetic composition as defined by claim 35, comprising an anhydrous or aqueous solid or paste, emulsion, suspension or dispersion.

37. The sunscreen or cosmetic composition as defined by claim 22, comprising a shampoo, lotion, gel, emulsion, vesicular dispersion, hair dye or bleach, hair spray, or rinse.

38. A method for protecting human skin, hair or both against the deleterious effects of ultraviolet irradiation, comprising topically applying thereto an

effective UV-photoprotecting amount of the sunscreen/cosmetic composition as defined by claim 22.

39. A method for protecting human skin, hair or both against the deleterious effects of solar radiation, comprising topically applying thereto an effective UV-photoprotecting amount of the sunscreen/cosmetic composition as defined by claim 22.